

**1999 Performance Report
General Chemistry and
Microbiology Analyses Section**

November 2000

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1999 PERFORMANCE REPORT
GENERAL CHEMISTRY AND MICROBIOLOGY SECTION

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Laboratory Services Branch

Ontario Ministry of the Environment

November 2000

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INTRODUCTION

The General Chemistry and Microbiology Section (GCMS) is part of the Ministry of the Environment Laboratory Services Branch. The section is comprised of three units, two of these, Water Chemistry and Dorset, identify and provide quantitative analysis for major ions, nutrients, and physical properties in a variety of matrices. The Microbiology Unit identifies and enumerates indicator bacteria of water and waste waters.

This report provides a brief outline of the analytical quality control (QC) program associated with sample analysis and examines 1999 performance data for each test in the Water Chemistry and Dorset Units. GCMS strives to maintain a high standard of analytical performance through its quality assurance program. QC is an integral part of this process.

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1.0 PERFORMANCE REPORT FORMAT

The parameters are those analysed by the GCMS for 1999.

The performance report is organized alphabetically according to test name (eg. Dissolved Organic Carbon is filed under the heading "Carbon, Dissolved Organic") and second, by the method reference number. Detailed information concerning the format of each page is outlined below:

1.1 TEST DESCRIPTION

<u>TITLE:</u>	The name of the test parameter.
<u>IDENTIFICATION:</u>	
Laboratory	Location where the test is performed.
Method Reference No:	A number assigned by the Quality Management Unit to an analytical test method eg.(E3370).
Product Code:	LIMS code for analysis request.
Sample Type/Matrix:	The various sample types that can be routed to the method.
Method Introduced:	Date that the method was implemented at the laboratory.
Reporting Units:	Unit of measurement in which the results are reported.
Supervisor:	Name of supervisor/manger responsible for the method.

SAMPLING:

The type of container and preservative (if applicable) that is used and minimum volume of sample that is usually required. Any sample preparation that is normally performed in the field, is also indicated (1).

SAMPLE PREPARATION:

Sample preparation techniques which are usually performed at the laboratory before analysis.

ANALYTICAL PROCEDURE:

Brief summary of the analytical method used to determine the parameter.

INSTRUMENTATION:

Type of instrumentation used to perform the test. Automated continuous flow systems consist of a sampler, peristaltic pump, manifold for reagent addition, detection system and readout system. Microcomputers are used to control the operation of analytical equipment and/or data acquisition.

REPORTING:

W and T are low level data qualifiers (2). A value reported as $\leq W$ is interpreted as not present, the value accompanying the remark is the lowest reportable value of the method under routine operating conditions. A value (multiple of W) reported as $\leq WE$ is interpreted as above for $\leq W$ following non-routine dilution of the sample to allow analysis of the target substance. A value reported as $< T$ is interpreted as target substance identified, use caution in interpretation unless more sample data supports this result. A value reported as $< TE$ is interpreted as above for $< T$ following non routine dilution of the sample to allow analysis for the target substance.

To provide a consistent LSB approach to data reporting, GCMS calculates W from the standard deviation of duplicates (S_2), near zero, by rounding down to the nearest 1,2 or 5 digit (4). T is five times W. The latest calculations, valid at date of publication for W and T values of all active methods, are contained in this report (APPENDIX A).

Data is reported to a maximum of three significant figures. Low level results are reported in increments of "W".

CALIBRATION:

The number of standards used to calibrate the analytical system plus blanks if applicable.

CONTROLS:

The calibration, drift, recovery, and interference controls that are used when applicable to ensure that the system is operating properly.

MODIFICATIONS:

Modifications made to the test in 1999.

NOTES:

Explanatory notes which may aid the data user in interpreting results and information.

1.2 PERFORMANCE DATA SUMMARY

QUALITY CONTROL DATA FROM/TO:

The period of time over which data were collected.

ANALYTICAL RANGE AND REPORTING UNIT:

The full scale value for the analytical range is given in concentration units.

CALIBRATION CONTROL:

Calibration control includes a table outlining the number of data collected over the selected time period, expected concentrations of the control standards, the calculated mean concentration of these standards, mean bias (mean concentration minus the expected concentration), and standard deviations of each control standard. The between run standard deviation (S), the within run standard deviation (S_w), the ratio S/S_w , and the control limits for standards sums and differences are provided.

RECOVERIES (Where applicable):

The table outlines the number of data collected over the selected time period, expected concentrations of the recovery standards, the calculated mean concentration of these standards, and standard deviations of each recovery standard.

DUPLICATES:

The table outlines within run duplicate data collected over the selected time period. Data are sorted into a number of concentration spans. The standard deviation for duplicates is provided for each range. The coefficient of variation (%) is determined by dividing the mean standard deviation (S_2) for a particular concentration span by the mean concentration of duplicate results in that span and multiplying by 100.

OTHER CHECKS (Where applicable):

The table outlines the number of data collected over a selected time period, the calculated mean concentration of ie., blank, and standard deviation.

1.3 QUALITY CONTROL GRAPHICS

CALIBRATION CONTROL:

Calibration control standards sums and differences are plotted on a horizontal scale for the period of data collection (referred to on the graphs as "QUALITY CONTROL STANDARD A+B" for example). The vertical scale consists of the warning/control limits expressed on either side of the expected value. These limits were chosen from analytical performance data.

2.0 ANALYTICAL QUALITY CONTROL PROGRAM

Quality control is a continuous process that involves constant checks of sample processing procedures. This report summarizes the QC data collected during analytical processing to monitor performance of the analytical system.

Calibration Standards are verified for identity, purity and concentration accuracy by comparison against independent sources wherever possible. A series of calibration standards are analyzed covering the analytical range. Calibration curve characteristics (slope, intercept and curvature) are examined on a per run basis as indicated by the method.

Once a system has been calibrated, quality control begins. Depending on the analytical procedure, quality control may be used to evaluate: calibration, blank, recovery, sensitivity, potential interference, and sample repeatability.

Calibration and Blank

Calibration is controlled by a minimum of two quality control standards and a long term blank which are prepared and maintained independently of the calibration standards. The system is not calibrated with the quality control standards. The long term blank is Pure De-ionized Water (Pure-DW) used to prepare the quality control standards and has zero concentration of the target analyte. Control standards are prepared less frequently than calibration standards and errors in newly prepared calibration standards can be detected by this cross check. Newly prepared control standards are run in parallel with in-use control standards and must meet control requirements over three consecutive runs before the new standards are accepted for routine use.

The standard deviation of the control standards is used to estimate the between-run standard deviation (S) and is compared against the within-run standard deviation (S_w). If the ratio S/S_w exceeds 1.5 then poor control of systematic error can be inferred (3). Values for S and S_w are calculated as follows:

$$2S^2 = (S_A)^2 + (S_B)^2 \qquad 2S_w^2 = (S_{A-B})^2$$

Where

S_A = standard deviation (s.d.) of control standard A

S_B = s.d. of control standard B

S_{A-B} = s.d. of the difference between control standards A and B

NOTE: If a second range is employed for a test, more control standards are used because, in many systems, the between-run standard deviations are concentration dependent.

Detailed description of the quality control processes are outlined in several LSB documents (2)(4)(5) and (6).

The control standards data are assessed and compared against the control/warning limits established from previous data to determine whether the calibration process is in control. The limits are set up initially based on method performance(4), and are reviewed when method and/or performance data reviews are conducted to determine if modifications are required based on historical data calculations. Control limits are calculated for the sums and differences of control standards (A,B,C,D) by the equations:

$(A+B) \pm 4(S_{A-B})$ for the sum of A+B

$(B+C) \pm 4(S_{B-C})$ for the sum of B+C

$(C+D) \pm 4(S_{C-D})$ for the sum of C+D

$(A-B) \pm 3(S_{A-B})$ for the difference of A-B

$(B-C) \pm 3(S_{B-C})$ for the difference of B-C

$(C-D) \pm 3(S_{C-D})$ for the difference of C-D

Note: Warning Limits are calculated by the same formulae above (using ± 2 instead of 4 and 3 respectively).

If a control limit is exceeded, the analysis is stopped, corrective action taken and the control standards are re-analysed.

Recovery

Some methods require sample pre-treatment, such as digestion or extraction. A recovery check, suitable to that method, is required to estimate the efficiency of the pre-treatment. Recovery standards are usually prepared at 0%, 20% and 80% of full scale. The solutions are analysed in the same manner as routine samples. Although these solutions are not used to calibrate the instrument, corrections for the blank and matrix effects are calculated and applied if necessary. For an analytical run to be accepted, the recoveries should be within $\pm(5\% + T/2)$ of their expected values. (See Section 1.1 "Reporting" for T determination). The average blank should be within three standard deviations of its historical mean. If a second range is employed for a test, at least one additional recovery standard is used.

Sensitivity and Baseline

Any change in the sensitivity of the instrumentation is monitored periodically during the run, as defined by the method, by analysing a standard that is usually 80% of full scale, and comparing the reading to the original calibration standards. Baseline drift is usually recorded by periodic analysis, as defined by the method, of Pure-DW which does not contain any of the analyte, but may be treated to correspond to sample pre-treatment.

Interference

The interference check is run on any test where a substance may be present in concentrations that affect the results. The check is carried out near the threshold concentration of the interfering substance, beyond which the methodological safeguards used to minimize the interference are no longer effective. The check indicates that the interference has no effect up to the specified concentrations.

Sample Repeatability

Generally, one sample out of twenty is analysed in duplicate up to a maximum of three duplicates per analytical run. The samples are selected for non-adjacent, within-run duplicate analyses. By analysing samples in duplicate, the ability of the analyst to obtain repeatable analytical results, within an analytical run, can be determined. For results to be acceptable, at least two of the three duplicate pairs must conform to limits that are set based on historical performance.

The observed differences in duplicate results are accumulated and sorted into 3 to 4 ranges based on analyte concentration. A standard deviation is calculated for each concentration range. The algorithm differs from the conventional standard deviation as follows:

Conventional Std. Dev. (1)*

$$S_1 = \sqrt{\frac{\sum_{i=1}^n (\bar{x} - x_i)^2}{n-1}}$$

Std. Dev. of Duplicates (2)*

$$S_2 = \sqrt{\frac{\sum_{i=1}^{n'} (x_1 - x_2)_i^2}{2n'}}$$

* Standard deviations used for the data summaries.

Where

S_1 = sample standard deviation

S_2 = duplicate difference standard deviation

n = number of data

\bar{x} = mean of data

x_i = i^{th} result

$(x_1 - x_2)_i$ = difference of the i^{th} duplicate

n' = number of duplicate pairs

The standard deviation (S_2) of the duplicate difference is also expressed as the coefficient of variation (CV).

$$CV = \frac{S_2}{\bar{X}} \times 100$$

2.1 PERFORMANCE SUMMARIES

ACIDITY, GRAN

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/08/82
Method Reference No	E3248	Reporting Units	µg/L as H ⁺
LIMS Product Code	PHACD3248	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Raw Sewage, Drinking Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or Plastic

ANALYTICAL PROCEDURE:

Sample aliquots (50.0 mL) are titrated with 0.01 N sodium hydroxide to pH >8.3. The titrant is standardized against 0.0005 N potassium hydrogen phthalate. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH readings following each aliquot of titrant. Data are subjected to Gran analysis.

pH and total fixed endpoint acidity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 5	Current T value: 25
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CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration	Long Term Blank plus 2 standards, e.g. QCA
-------------	--

NOTES:

Qct '99. The W value of 1.0 and T value of 5.0 were changed to 5.0 and 2.0 respectively based on historical data.

ACIDITY, GRAN (E3248)

QUALITY CONTROL DATA FROM 02/02/99 TO 07/12/99

Analytical Range: to 1000 µeq/L as H⁺

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	16	500.0	499	-1	8.5905
B:	16	200.0	198	-2	8.0289
A+B:		700.0	697	-3	14.2027
A-B:		300.0	301	1	8.6487

s.d.(AB)

S(between runs): 5.9

Sw(within run): 4.3

S/Sw:1.4

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

662 - 738 for A+B
272 - 328 for A-B

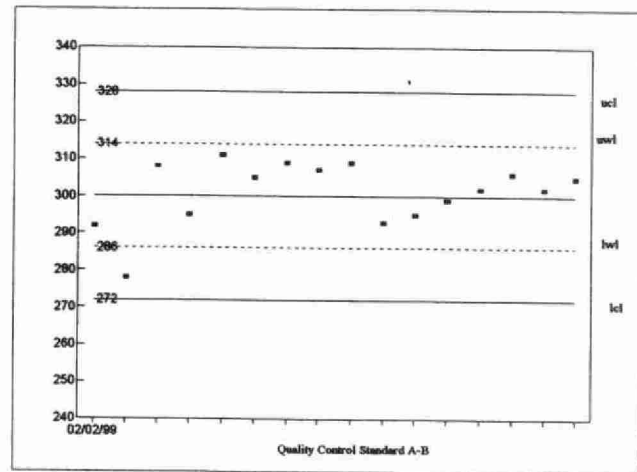
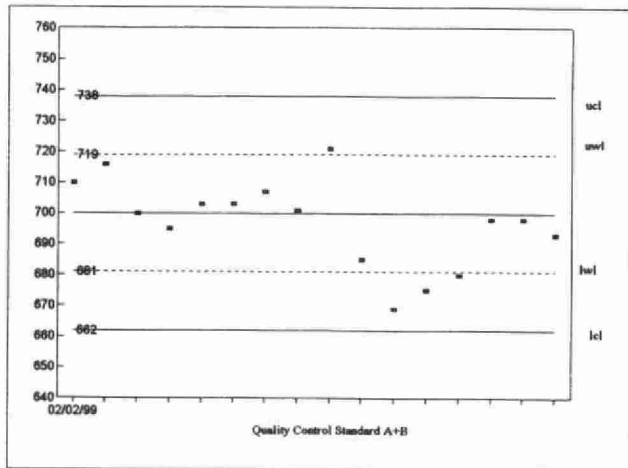
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
25	0 - 100	4.0497	7.4
10	101 - 250	6.4653	5.3
0	250 - 500	N.A.	N.A.
35	Overall	4.8639	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	16	-1.7	4.7387

ACIDITY, GRAN ($\mu\text{eq/L}$ as H^+)
QUALITY CONTROL DATA FROM 02/02/99 To 07/12/99
E3248



ACIDITY, TOTAL FIXED ENDPOINT

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/08/82
Method Reference No.	E3248	Reporting Units	mg/L as CaCO ₃
LIMS Product Code	PHACD3248	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Raw Sewage, Drinking Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or Plastic

ANALYTICAL PROCEDURE:

Sample aliquots (50.0 mL) are titrated with 0.01 N sodium hydroxide to pH >8.3. The titrant is standardized against 0.0005 N potassium hydrogen phthalate. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH readings following each aliquot of titrant. pH and gran acidity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1.0
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CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
-------------	---------------------------------

NOTES:

Qct '99. The W value of 0.05 and T value of 0.25 were changed to 0.2 and 1.0 respectively based on historical data.

ACIDITY, TOTAL FIXED ENDPOINT (E3248)

QUALITY CONTROL DATA FROM 02/02/99 TO 07/12/99

Analytical Range: to 100 mg/L as CaCO₃

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	16	25.0	24.91	-0.09	0.4325
B:	16	10.0	9.79	-0.11	0.4060
A+B:		35.0	34.68	-0.32	0.7340
A-B:		15.0	15.13	0.13	0.4070

s.d.(AB)

S(between runs): 0.297

Sw(within run):0.204

S/Sw: 1.5

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

32.8 - 37.2 for A+B
13.4 - 16.6 for A-B

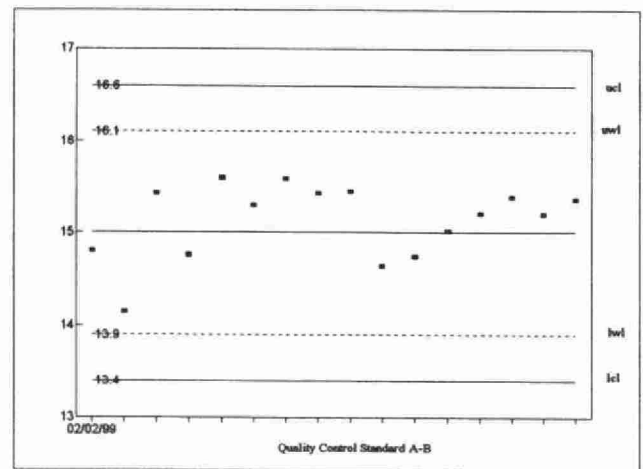
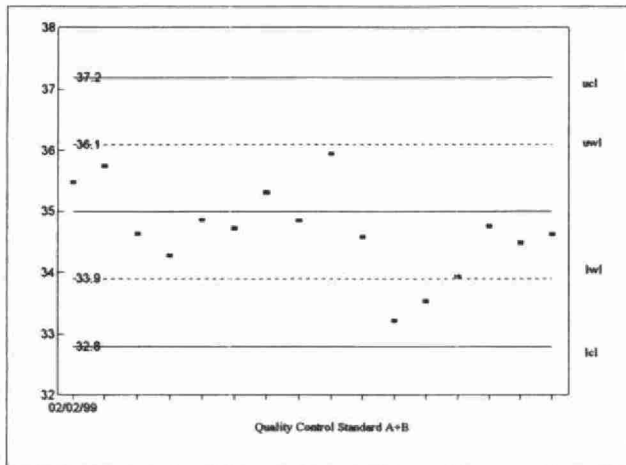
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
17	0.0 - 4.0	0.0799	3.2
19	4.1 - 10.0	0.2303	4.1
0	10.1 - 20.0	N.A.	N.A.
37	Overall	0.1761	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	16	0.075	0.1661

ACIDITY, TOTAL FIXED ENDPOINT (mg/L as CaCO₃)
 QUALITY CONTROL DATA FROM 02/02/99 TO 07/12/99
 E3248



ALKALINITY, GRAN

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	26/07/79
Method Reference No.	E3042	Reporting Units	mg/L as CaCO ₃
LIMS Product Code	PHALK3042	Manager	W. Scheider
Sample Type/Matrix:	Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required:	150 mL
Container:	250 mL Amber polyethylene bottle filled to the brim; screw caps with cone-shaped liners are preferred.

ANALYTICAL PROCEDURE:

Samples (50 mL) are weighed (volume = weight), and titrated with 0.02 N sulphuric acid to a pH <3.7. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant. Data are subjected to Gran analysis. pH and Alkalinity, Total Fixed Endpoint are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data reduction software.

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7

CONTROLS:

Calibration	LTBL plus 4 standards, e.g. QCA
Drift	2 standard buffers - 2 times daily

ALKALINITY, GRAN (E3042)

QUALITY CONTROL DATA FROM 08/01/99 TO 17/12/99

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	71	20.0	19.39	-0.61	0.2590
B:	71	5.0	4.67	-0.33	0.4008
C:	71	-5.0	-5.11	-0.11	0.8315
D:	71	-1.25	-1.73	-0.47	0.5347
A+B:		25.0	24.52	-0.48	0.5386
A-B:		15.0	14.72	-0.28	0.3260
C+D:		-6.25	-6.37	-0.12	0.8680
C-D:		-3.75	-3.39	0.36	0.9611

s.d.(AB) S(between runs): 0.34

Sw(within run): 0.23

S/Sw: 1.46

s.d.(CD) S(between runs): 0.70

Sw(within run): 0.68

S/Sw: 1.02

The calibration is accepted if the calibration control values obtained lie within the ranges:

24.0	-	26.0	for	A+B
14.0	-	16.0	for	A-B
-8.89	-	-3.61	for	C+D
-5.73	-	-1.77	for	C-D

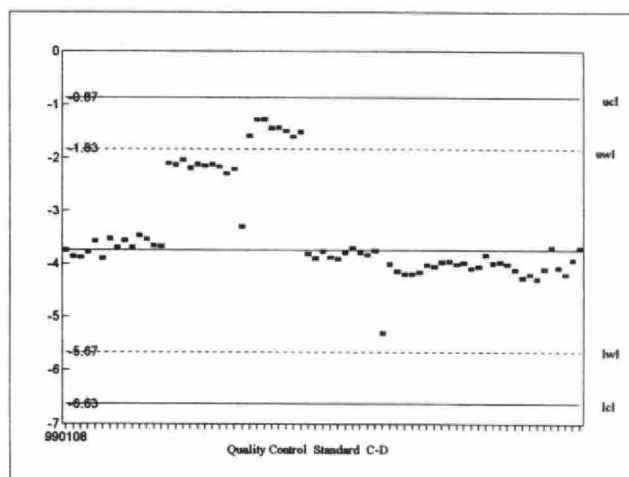
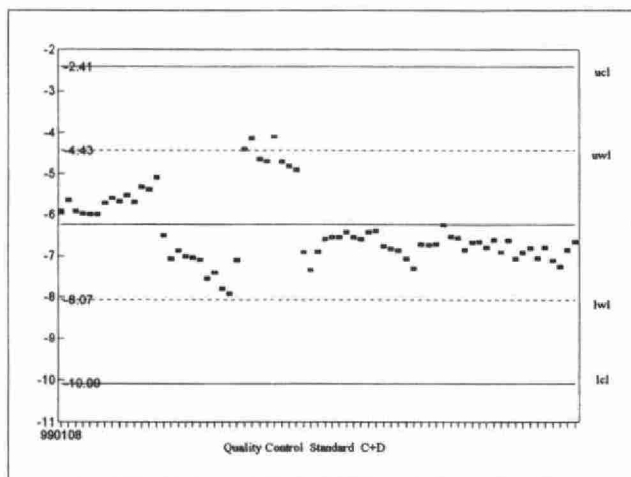
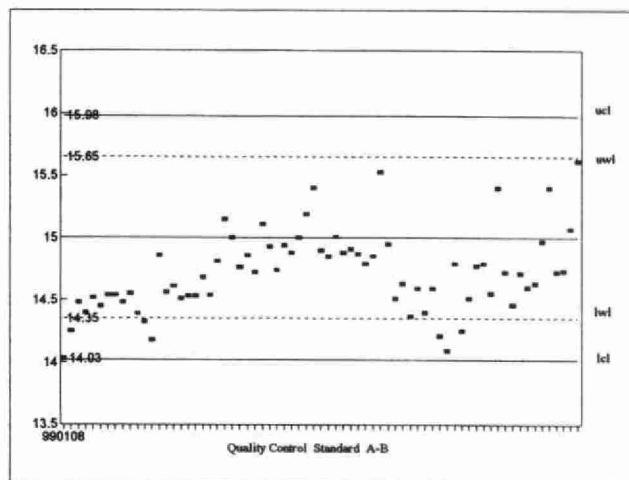
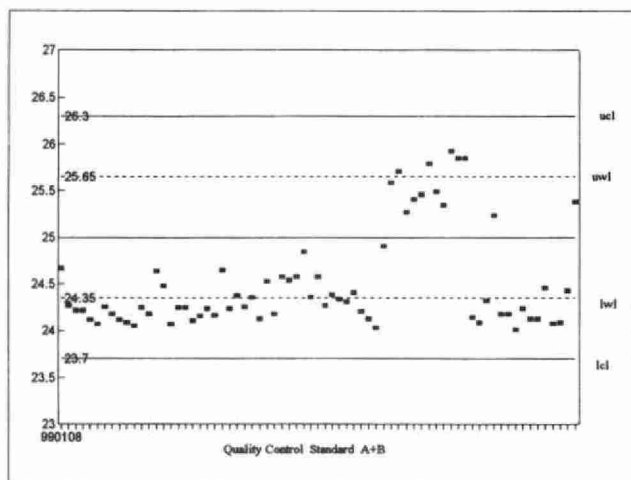
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
202	-5.0 - 30.0	0.2298	14.0
4	31 - 100	0.3294	0.5
3	100 - 300	0.7178	0.3
189	OVERALL	0.2460	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	71	-.2342	0.2133

ALKALINITY, GRAN QUALITY CONTROL DATA FROM 08/01/99 TO 17/12/99 E3042



ALKALINITY, TOTAL FIXED ENDPOINT

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	26/07/79
Method Reference No.	E3042	Reporting Units	mg/L as CaCO ₃
LIMS Product Code	PHALK3042	Manager	W. Scheider
Sample Type/Matrix:	Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required:	150 mL
Container:	250 mL Amber polyethylene bottle filled to the brim; screw caps with cone-shaped liners are preferred.

ANALYTICAL PROCEDURE:

Samples (100 mL) are weighed (volume = weight), and titrated with 0.02 N sulphuric acid to a pH 4.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant.

pH and Alkalinity, Gran are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.05	Current T value: 0.25
--------------------------------	-----------------------	-----------------------

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
Drift	2 standard buffers - once daily

ALKALINITY, TOTAL FIXED ENDPOINT (E3042)

QUALITY CONTROL DATA FROM 08/01/99 TO 17/12/99

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	62	20.0	20.93	0.93	0.3875
B:	62	5.00	6.45	1.45	0.4765
A+B:		25.0	24.57	-0.43	0.5719
A-B:		15.0	14.53	-0.47	0.3139

s.d.(AB) S(between runs): 0.43

Sw:(within run): 0.22

S/Sw: 2.0

On any given day the calibration is accepted if the values obtained lie within the ranges:

24.0 - 26.0 for A+B
14.0 - 16.0 for A-B

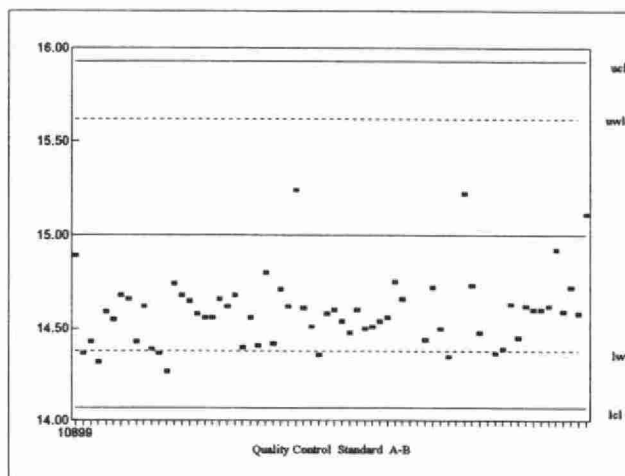
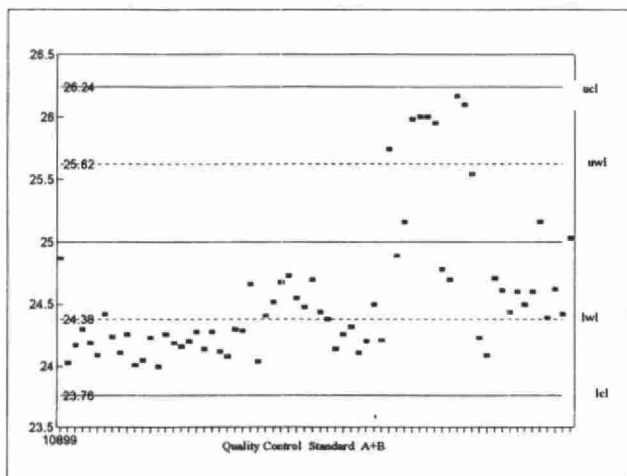
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
159	0.0 - 30	0.0860	1.8
4	31 - 150	0.3997	0.5
3	151 - 300	0.9032	0.4
166	Overall	0.1601	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	68	1.4026	0.3063

ALKALINITY, TOTAL FIXED ENDPOINT (mg/L as CaCO₃)
 QUALITY CONTROL FROM 08/01/99 TO 17/12/99
 E3042



ALKALINITY, TOTAL FIXED ENDPOINT

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	09/07/80
Method Reference No.	E3218	Reporting Unit	mg/L as CaCO ₃
LIMS Product Code	PHALCO3218	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Effluent, Industrial Waste, Raw Sewage, Drinking Water, Ground Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or Plastic

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.02 N sulphuric acid to pH endpoint of 4.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant.

pH, and conductivity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.5	Current T value: 2.5
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CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration	BL plus 4 standards, e.g. QCA
Drift	In run standards throughout the run (tap water diluted to 50% V/V)

NOTES:

May '97 the W value was changed from 0.2 to 0.5 after a review of 2 years low level duplicate data '94-95.

ALKALINITY, TOTAL FIXED ENDPOINT (E3218)

QUALITY CONTROL DATA FROM 05/01/99 TO 29/12/99

Analytical Range: to 1000 mg/L as CaCO₃

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	107	250	249.58	-0.42	1.8442
B:	107	100	99.83	-0.17	0.8758
C:	107	25	25.11	0.11	0.8991
D:	107	2.5	2.48	-0.02	0.1352
A+B:		350	349.42	-0.58	2.2609
A-B:		150	149.76	-0.24	1.7956
B+C:		125	124.94	-0.06	1.3407
B-C:		75	74.72	-0.28	1.1633
C+D:		27.5	27.59	0.09	0.8974
C-D:		22.5	22.20	-0.30	3.2123

s.d.(AB) S(between runs): 1.44

Sw(within run): 1.27

S/Sw: 1.1

s.d.(BC) S(between runs): 0.89

Sw(within run): 0.82

S/Sw: 1.1

s.d.(CD) S(between runs): 0.64

Sw(within run): 2.27

S/Sw: 0.3

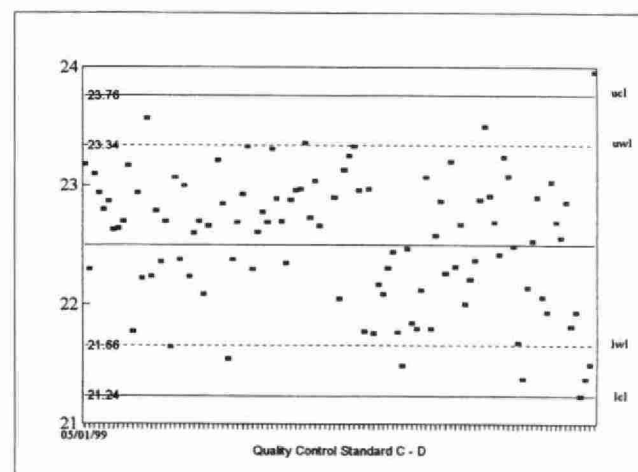
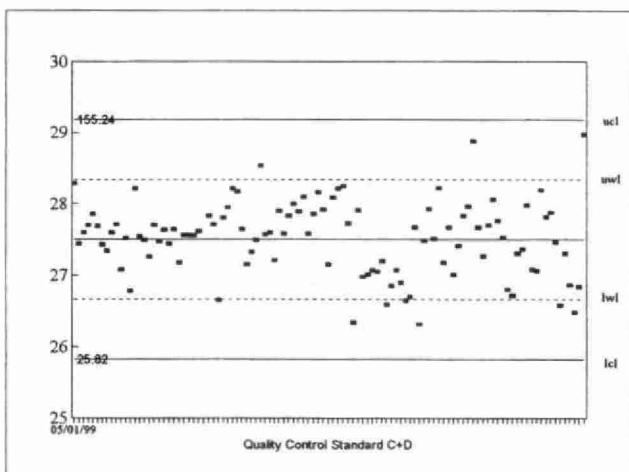
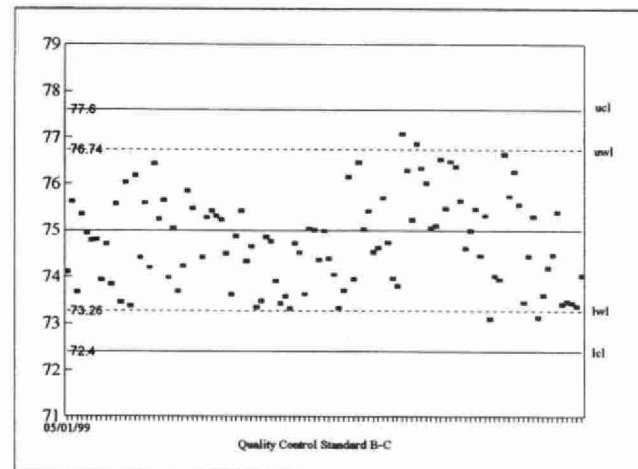
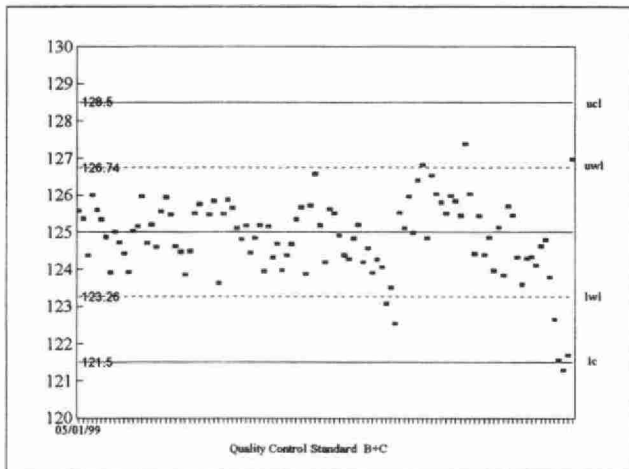
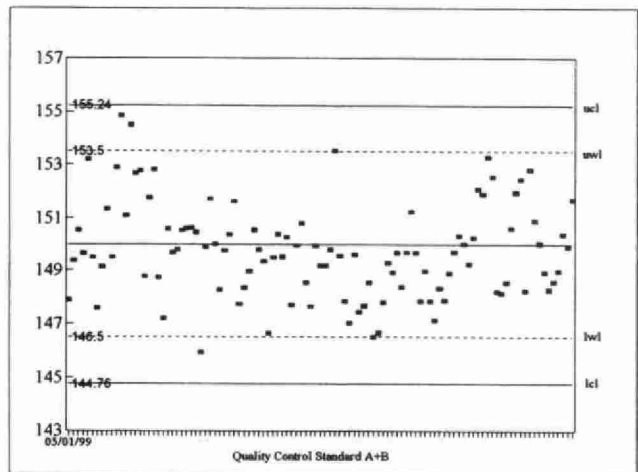
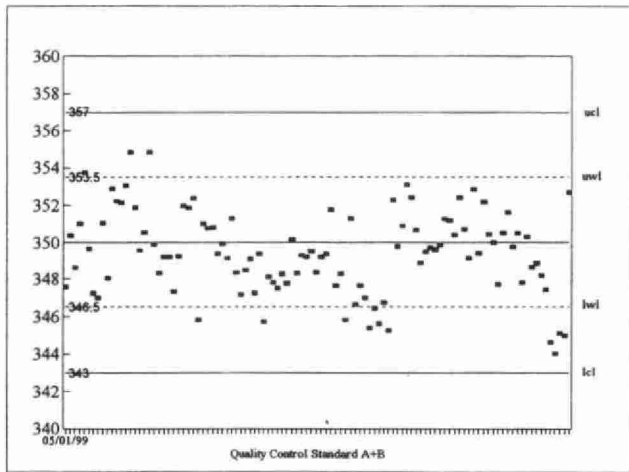
On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

343	-	357.0	for	A+B
144.76	-	155.24	for	A-B
121.5	-	128.5	for	B+C
72.4	-	77.6	for	B-C
25.82	-	29.18	for	C+D
21.24	-	23.76	for	C-D

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
165	0 - 50	0.4147	1.0
58	51 - 100	0.7147	0.5
68	101 - 300	0.8550	0.8
1	301 - 1000	N.A.	N.A.
1	>1000	N.A.	N.A.
293	Overall	0.5732	

ALKALINITY, TOTAL FIXED ENDPOINT (mg/L as CaCO₃)
 QUALITY CONTROL DATA FROM 05/01/99 TO 29/12/99
 E3218



ALUMINUM, TOTAL

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	06/09/83
Method Reference No.	E3300	Reporting Units	µg/L as Al
LIMS Product Code	AL3300	Manager	F. Tomassini
Sample Type/Matrix	Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	10 mL
Container	100 mL Polypropylene bottle capped, acidified to 0.1% with HNO ₃

ANALYTICAL PROCEDURE:

This procedure is based on the formation of an aluminum-catechol violet complex at pH 6.2. Acidified samples are oxidized by UV digestion for total aluminum. Phenanthroline-hydroxylamine-HCL reagents are used to reduce interference by iron. Concentrations of aluminum are determined by comparison with a similarly prepared series of standards.

INSTRUMENTATION:

UV-digestor
An autoanalyzer with microprocessor for DCI system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 2	Current T value: 10
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CALIBRATION:

BL plus 8 standards daily

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL every 10 samples and BL plus check standard every 20 samples

NOTES:

Method was discontinued in Feb 1999. Analysis for Aluminum was transferred to method E3386 under the LIMS product code MET3386.

CALCIUM

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	20/07/88
Method Reference No.	E3249	Reporting Unit	mg/L as Ca
LIMS Product Code	CAT3249	Supervisor	F. Tomassini
Sample Type/Matrix	Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	5 mL
Container	Plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 422.7 nm with an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.
Approximate absorbance: 0.2 at the full scale level.

INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.02	Current T value: 0.1
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CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g., QCA
Drift	BL, reslope standard every 10 samples.

CALCIUM (E3249)

QUALITY CONTROL DATA FROM 12/01/99 TO 22/12/99

Full Scale: to 8.0 mg/L as Ca

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	47	6.4	6.437	0.037	0.0531
B:	47	1.6	1.605	0.005	0.0205
C:	47	0.4	0.402	0.002	0.0170
A+B:		8.0	8.042	0.042	0.0684
A-B:		4.8	4.832	0.032	0.0425
B+C:		2.0	2.007	0.007	0.0335
B-C:		1.2	1.203	0.003	0.0172

s.d.(AB) S(between runs): 0.040 Sw(within run): 0.030 S/Sw: 1.34
s.d.(BC) S(between runs): 0.012 Sw(within run): 0.012 S/Sw: 0.99

The calibration is accepted if the calibration control values obtained lie within the ranges:

7.828 - 8.172 for A+B
4.671 - 4.929 for A-B
1.937 - 2.063 for B+C
1.153 - 1.247 for B-C

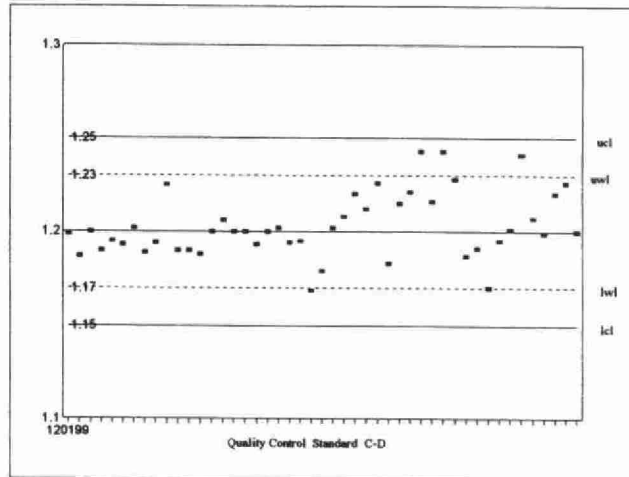
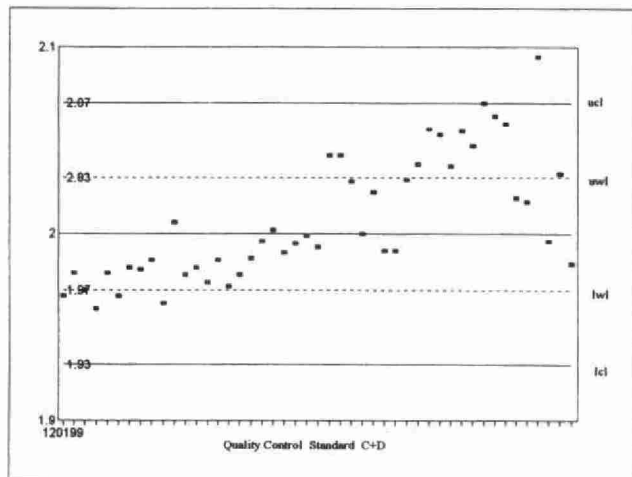
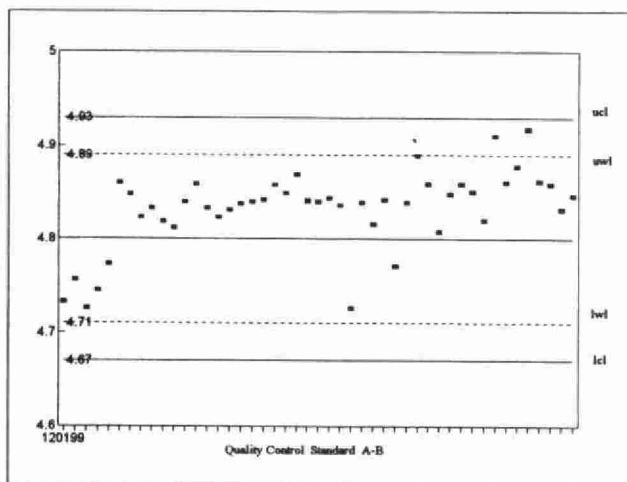
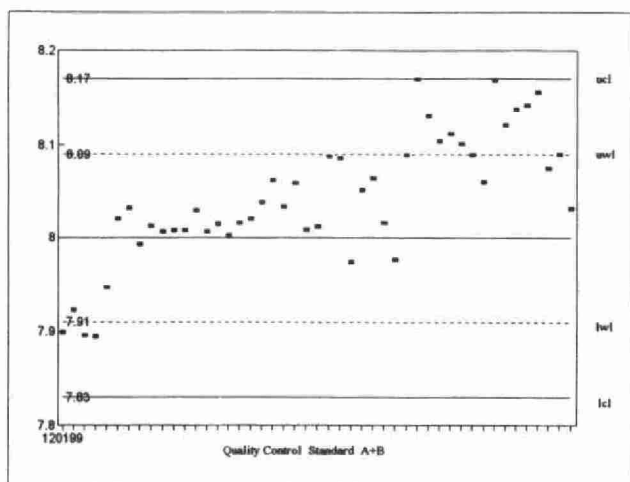
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
35	0.00 - 0.80	0.0246	7.2
32	0.81 - 1.60	0.0241	1.7
98	1.61 - 4.00	0.0630	2.5
12	4.01 - 8.00	0.0546	1.0
177	Overall	0.0512	2.6

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	47	-0.0203	0.1204

CALCIUM (mg/L as Ca)
QUALITY CONTROL DATA FROM 12/01/99 TO 22/12/99
E3249



CARBON, DISSOLVED INORGANIC

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	03/06/80
Method Reference No.	E3028	Reporting Unit	mg/L as C
LIMS Product Code	CARB3028	Supervisor	F. Tomassini
Sample Type/Matrix	Leachate, Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass

ANALYTICAL PROCEDURE:

Dissolved inorganic carbon, which is determined colourimetrically on the supernatant of a settled sample, is converted to carbon dioxide gas by acidification. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved inorganic carbon content of the sample.

Approximate absorbance: 0.3 at the full scale level.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: air (CO₂-free) supply, dialysis unit. Colourimetric measurement is through a 5.0 cm. light path at 550 nm.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.02	Current T value: 0.1
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CALIBRATION:

BL plus 9 standards daily

CONTROLS:

Calibration	LTB plus 3 standards, e.g. QCA, QCB, QCC
Drift	BL every 10 samples; BL plus 1 check standard every 20 samples

CARBON, DISSOLVED INORGANIC (E3028)

QUALITY CONTROL DATA FROM 11/01/99 TO 17/12/99

Full Scale: to 10.0 mg/L as C

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	38	8.0	7.91	-0.09	0.1071
B:	38	4.0	3.94	-0.06	0.0751
C:	38	0.8	0.72	-0.08	0.0422
A+B:		12.0	11.84	-0.16	0.1488
A-B:		4.0	3.97	-0.03	0.1100
B+C:		4.8	4.65	-0.15	0.0861
B-C:		3.2	3.22	0.02	0.0863

s.d.(AB) S(between runs):0.093

Sw(within run): 0.078

S/Sw: 1.2

s.d.(BC) S(between runs):0.061

Sw(within run): 0.061

S/Sw: 1.0

The calibration is accepted if the calibration control values obtained lie within the ranges:

11.4	-	12.6	for	A+B.
3.6	-	4.4	for	A-B
4.4	-	5.2	for	B+C
2.9	-	3.5	for	B-C

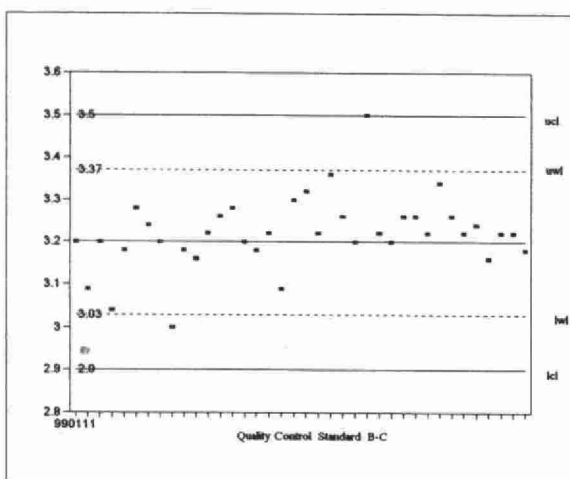
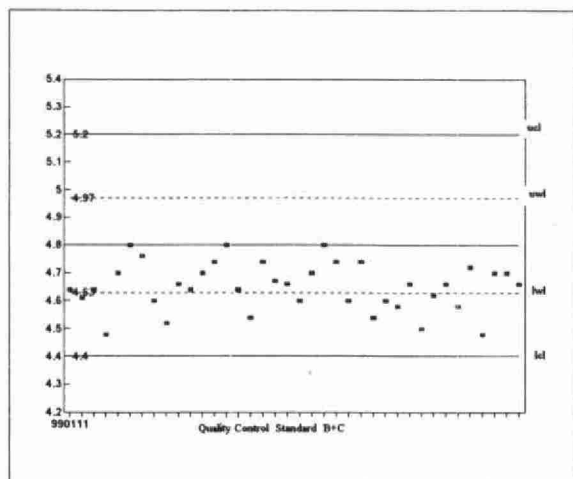
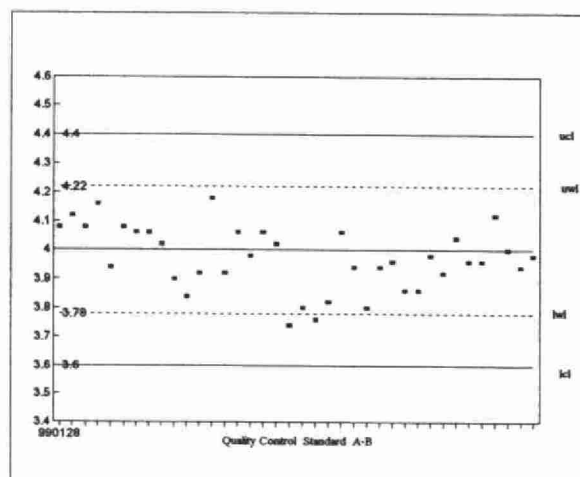
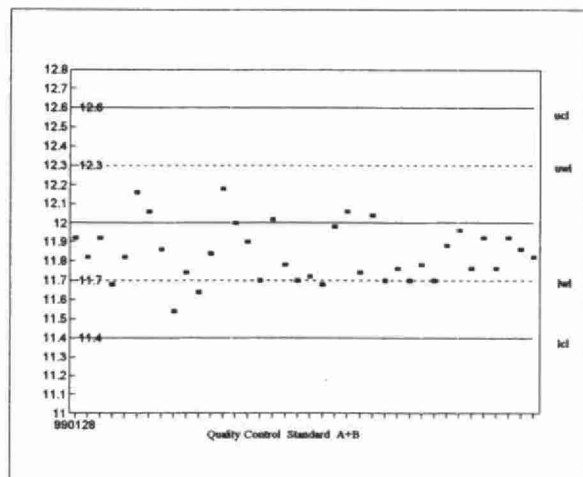
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
27	0.00 - 1.00	0.0166	2.3
35	1.01 - 2.00	0.0449	3.0
29	2.01 - 5.00	0.0691	2.4
22	5.01 - 10.0	0.1526	2.0
113	Overall	0.0803	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	38	0.244	0.0330

CARBON, DISSOLVED INORGANIC (mg/L as C)
QUALITY CONTROL DATA FROM 11/01/99 TO 17/12/99
E3028



CARBON, DISSOLVED INORGANIC

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3370	Reporting Unit	mg/L as C
LIMS Product Code	DCSI3370	Supervisor	P.Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water Ground Water, Leachates, Precipitation, Surface Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Dissolved inorganic carbon, which is determined colourimetrically on the supernatant of a settled sample, is converted to carbon dioxide gas by acidification. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved inorganic carbon content of the sample.

Approximate absorbance: 0.3 at the full scale level.

Dissolved organic carbon, and reactive silicates are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: air (CO₂-free) supply, dialysis unit. Colourimetric measurement is through a 5.0 cm. light path at 550 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1.0
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g., QCA
Drift	BL , standard and BL every 10 samples

NOTES:

December 1998: The HP data capture/processing system was replaced by Labtronics.

CARBON, DISSOLVED INORGANIC (E3370)

QUALITY CONTROL DATA FROM 18/01/99 TO 17/12/99

Full Scale: to 80.0 mg/L as C

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	57	64.0	63.83	0.17	0.7716
B:	57	16.0	15.91	-0.09	0.3556
C:	57	4.00	4.05	0.05	0.2540
A+B:		80.0	79.74	-0.26	0.8999
A-B:		48.0	47.91	-0.09	0.7961
B+C:		20.0	19.96	-0.04	0.5132
B-C:		12.0	11.87	-0.13	0.3443

s.d.(AB)

S(between runs): 0.60

Sw(within run): 0.56

S/Sw:1.1

s.d.(BC)

S(between runs): 0.30

Sw(within run): 0.24

S/Sw:1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

78.06	-	81.94	for	A+B
46.55	-	49.45	for	A-B
19.11	-	20.86	for	B+C
11.35	-	12.65	for	B-C

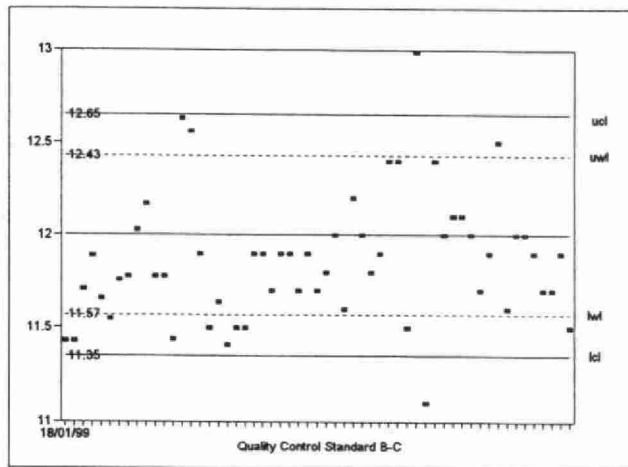
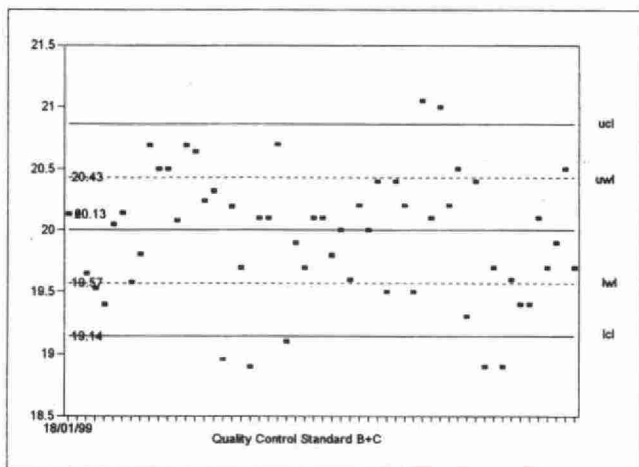
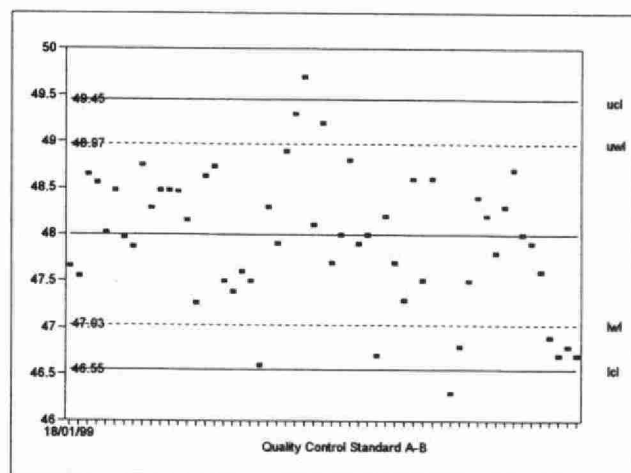
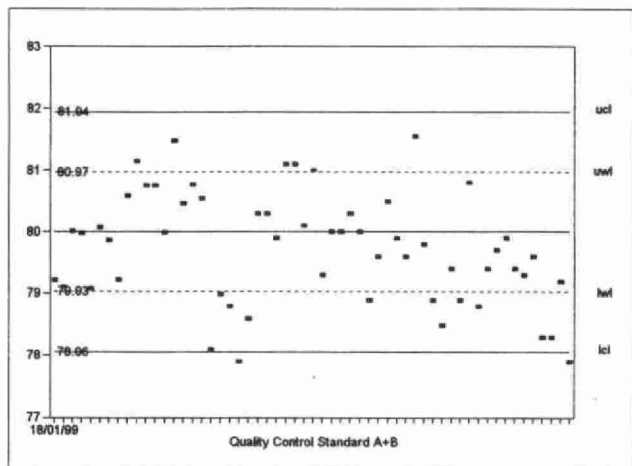
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
56	0.00 - 8.0	0.3491	24.9
11	8.1 - 16.0	0.3359	3.1
63	16.1 - 40.0	0.4218	1.8
14	40.1-80.0	0.7861	1.4
144	Overall		

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	57	0.1081	0.2747

CARBON, DISSOLVED INORGANIC (mg/L as C)
QUALITY CONTROL DATA FROM 18/01/99 TO 17/12/99
E3370



CARBON, DISSOLVED ORGANIC

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3370	Reporting Unit	mg/L as C
LIMS Product Code	DCSI3370	Supervisor	P.Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water Ground Water, Leachates, Precipitation, Surface Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Using an automated system, the supernatant from a settled sample is acidified and flushed with nitrogen gas to remove inorganic carbon. Organic carbon is then oxidized to carbon dioxide gas by exposure to ultra-violet light (UV) in acid-persulphate media. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved organic carbon content of the sample.

Approximate absorbance: 0.3 at the full scale level.

Dissolved inorganic carbon, and reactive silicates are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: nitrogen and air (CO₂-free) supplies with flow controls, dialysis unit, UV digester. Colourimetric measurement is through a 5.0 cm. light path at 550 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.1	Current T value: 0.5
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g., QCA
Drift	BL, standard and BL every 10 samples

NOTES:

December 1998: The HP data capture/processing system was replaced by Labtronics.

CARBON, DISSOLVED ORGANIC (E3370)

QUALITY CONTROL DATA FROM 18/01/99 TO 17/12/99

Full Scale: to 20.0 mg/L as C

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	57	16.0	16.04	0.04	0.1402
B:	57	4.00	3.98	-0.02	0.1293
C:	57	1.00	0.99	-0.01	0.1348
A+B:		20.0	20.03	0.03	0.1971
A-B:		12.0	12.06	0.06	0.1841
B+C:		5.00	4.97	-0.03	0.2208
B-C:		3.00	2.99	-0.01	0.1449

s.d.(AB) S(between runs): 0.1338

Sw(within run): 0.1304

S/Sw: 1.0

s.d.(BC) S(between runs): 0.1330

Sw(within run): 0.1030

S/Sw: 1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

19.44	-	20.56	for	A+B
11.58	-	12.42	for	A-B
4.72	-	5.28	for	B+C
2.79	-	3.21	for	B-C

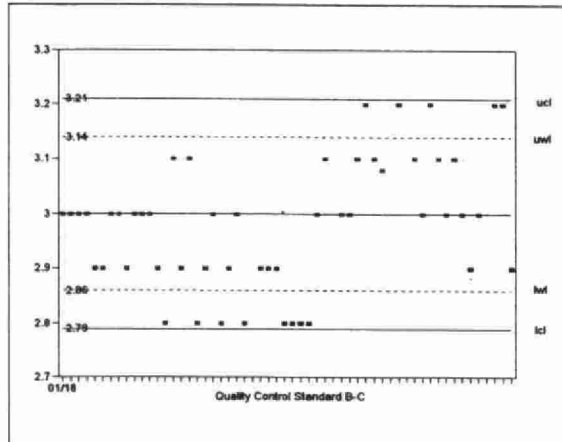
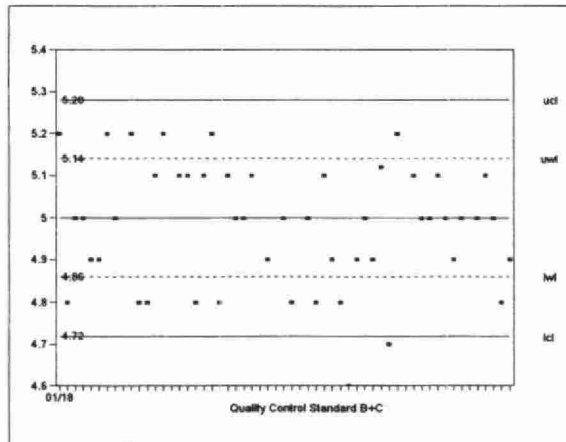
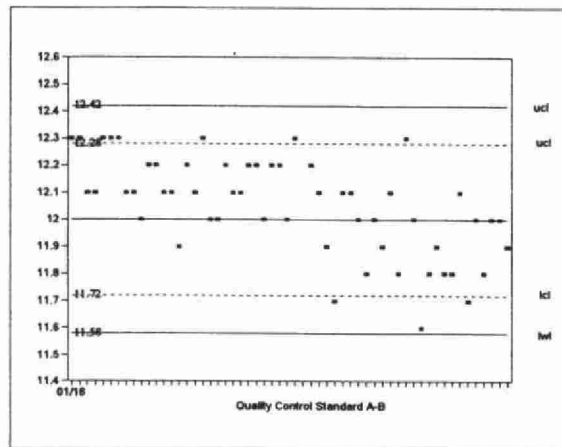
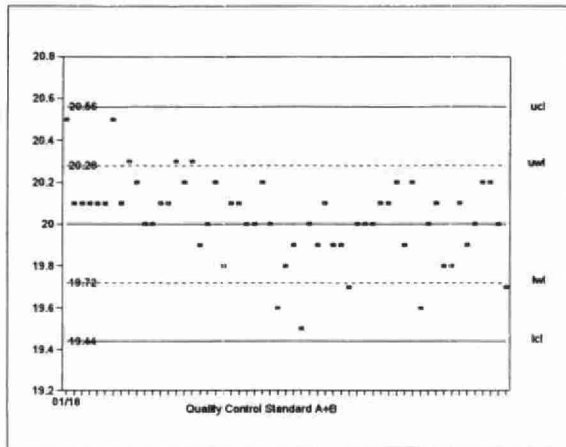
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
52	0.00 - 2.00	0.1105	7.3
43	2.01 - 4.0	0.1249	4.1
58	4.01 - 10.0	0.2305	3.9
9	10.01 - 20.0	0.2108	1.7
162	Overall	0.1719	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	57	-0.0536	0.2272

CARBON. DISSOLVED ORGANIC (mg/L as C)
QUALITY CONTROL DATA FROM 18/01/99 TO 17/12/99
E3370



CHLORIDE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3004	Reporting Unit	$\mu\text{g}/\text{m}^3$ as Cl
LIMS Product Code	ANION3004	Supervisor	P. Wilson
Sample Type/Matrix	Air; HiVol Glass Fibre, Quartz and Polyflon, Other Filters and Puff		

SAMPLING:

Quantity Required	3/4" or 1.9cm strip from 8"x10" filter
Container	50 mL polypropylene tube

SAMPLING PREPARATION:

A 3/4" filter strip is cut in pieces and deposited into a 50 mL polypropylene tube. 50 mL of Pure-Water is added to the tube. The tube is placed on a horizontal shaker for approximately 1 hour. The supernatant is then filtered into a 15 mL plastic tube and analysed.

ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of sodium bicarbonate and sodium carbonate and a conductivity detector. The concentration of chloride (mg/L) is determined by the comparison of the analyte peak area count to that of a series of standards. The analyte result is corrected for the filter blank before the final calculation. The result is reported as $\mu\text{g}/\text{m}^3$ as Cl.

Nitrate and sulphate are determined simultaneously.

INSTRUMENTATION:

Horizontal Shaker, ion chromatographic system plus a PC with ChromPerfect software and DT2804 card for automated sample injection, timing, and data processing.

REPORTING:

Maximum Significant Figures: 2	Current W value: $0.1 \mu\text{g}/\text{m}^3$	Current T value: $0.5 \mu\text{g}/\text{m}^3$
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CALIBRATION:

6 standards

CONTROLS:

Calibration	MB, IS(n), CS1, and CS2
Drift	Duplicate plus 2 standards approximately every 20 samples

CHLORIDE cont'd

NOTES:

To convert unit from mg/L to $\mu\text{g}/\text{m}^3$, the final concentration of Cl in mg/L is multiplied by the following formula:

$$\text{Result (mg/L)} \times 50\text{mL} \times (63/6.75) / \text{air volume} = \mu\text{g}/\text{m}^3$$

Where: 63 is the area of the filter exposed and 6.75 is the sample aliquot area in square inch.

CHLORIDE (E3004)

QUALITY CONTROL DATA FROM 1997 TO 1999

Analytical Range: to 14.31 $\mu\text{g}/\text{m}^3$

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
196	0.0 - 2.86	0.030	0.7
3	2.89 - 7.15	0.147	0.1
0	7.18 - 14.31	N.A.	N.A.
199	Overall	0.035	

CHLORIDE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/01/86
Method Reference No.	E3013	Reporting Unit	µg/g as Cl
LIMS Product Code	ANION3013, CL3013	Supervisor	P. Wilson
Sample Type/Matrix	Soil and Sediment		

SAMPLING:

Quantity Required	20 g
Container	glass or plastic

SAMPLING PREPARATION:

A 3.0 g sample air dried, sieved soil or air dried sieved and ground sediment is placed in a 50 mL centrifuge tube and shaken with 30 mL Pure-DW for 1 hour on a shaker. Samples are centrifuged, membrane filtered and analyzed for chloride and sulphate by ion chromatography.

ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of sodium bicarbonate and sodium carbonate and a conductivity detector. The concentration of chloride (mg/L) is determined by the comparison of the analyte peak area count to that of a series of standards. The result is reported as µg/g as Cl.

Sulphate is determined simultaneously.

INSTRUMENTATION:

Horizontal Shaker, ion chromatographic system plus a PC with ChromPerfect Spirit software and DT2804 card for automated sample injection, timing, and data processing.

REPORTING:

Maximum Significant Figures: 2	Current W value: 0.5 µg/g	Current T value: 2.5 µg/g
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CALIBRATION:

8 standards

CONTROLS:

Calibration	IS(n) - certified reference check solution, CS1, CS2, - check standards, in house matrix check standards MC(n)
Drift	Duplicate plus 2 check standards approximately every 20 samples
Contamination	Method Blank (MB)
Interference	SP1 and SP2 (spike solutions) for positive and negative interferences

CHLORIDE (E3013)

QUALITY CONTROL DATA FROM 1998 TO 2000

Analytical Range: to 500 µg/g

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
39	0 - 100	1.6500	7.8
3	101 - 250	2.0367	1.1
2	251 - 500	N.A.	N.A.
44	Overall	1.7513	

CHLORIDE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/05/75
Method Reference No.	E3016	Reporting Unit	mg/L as Cl
LIMS Product Code	CL3016	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water, Ground Water, Leachate, Surface Water		

SAMPLING:

Quantity Required:	10 mL
Container:	Plastic

ANALYTICAL PROCEDURE:

Chloride ions are combined with mercuric thiocyanate releasing thiocyanate quantitatively. Thiocyanate then reacts with ferric ions to produce ferric thiocyanate (red), and the absorbance of the latter is measured colourimetrically.

Approximate absorbance: 0.5 at the full scale level.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 1.5 cm light path at 480 nm.

Data capture and processing via a multistage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1
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CALIBRATION:

BL plus 12 standards

CONTROLS:

Calibration:	LTBL plus 3 standards, e.g. QCA
Drift:	BL and standard after every 12 samples

NOTES:

April 1998: The HP data capture/processing system was replaced by Labtronics. Two additional Calibration standards were added at the low end of the curve.

CHLORIDE (E3016)

QUALITY CONTROL DATA FROM 01/01/99 TO 31/12/99

Laboratory Unit: Colourimetry

Full Scale: to 100 mg/L as Cl

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	55	75.00	75.02	0.02	0.338
B:	55	25.00	25.03	0.03	0.140
C:	55	5.00	4.90	0.10	0.116
A+B:		100.00	100.05	0.05	0.359
A-B:		50.00	49.99	0.01	0.372
B+C:		30.00	29.93	0.07	0.214
B-C:		20.00	20.13	0.13	0.143

s.d.(AB) S(between runs):0.26

s.d.(BC) S(between runs):0.13

Sw(within run): 0.26

Sw(within run): 0.10

S/Sw: 0.98

S/Sw: 1.27

The calibration is accepted if the calibration control values obtained lie within the ranges:

98.51 - 101.49 for A+B

48.88 - 51.12 for A-B

29.43 - 30.57 for B+C

19.57 - 20.43 for B-C

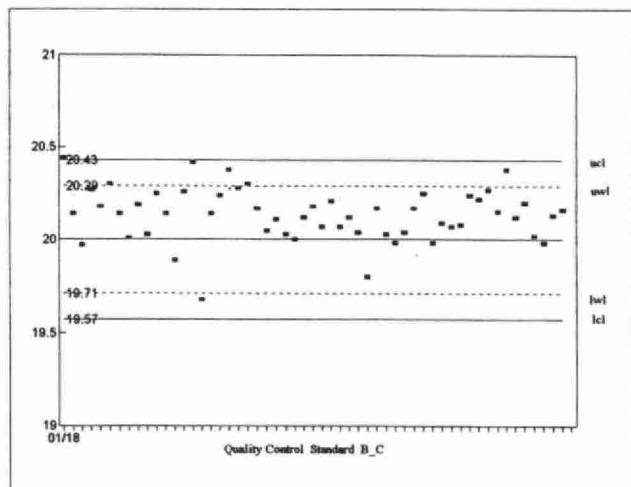
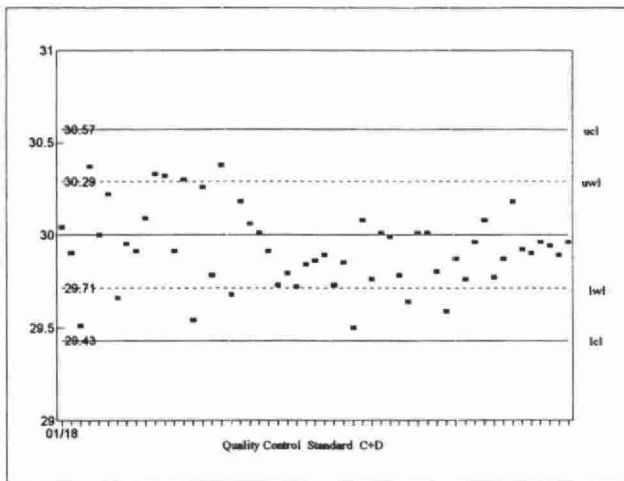
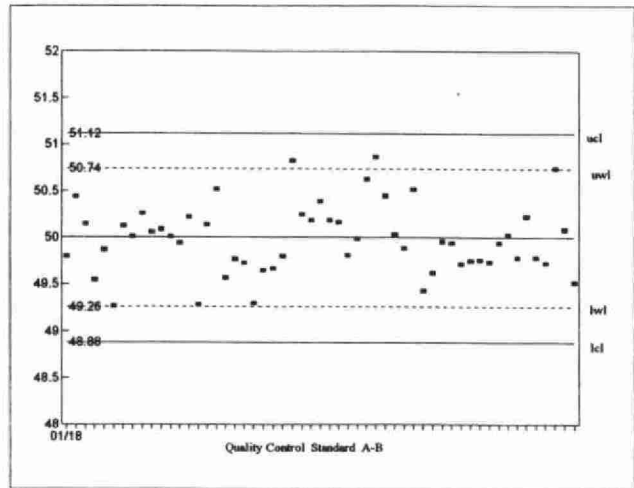
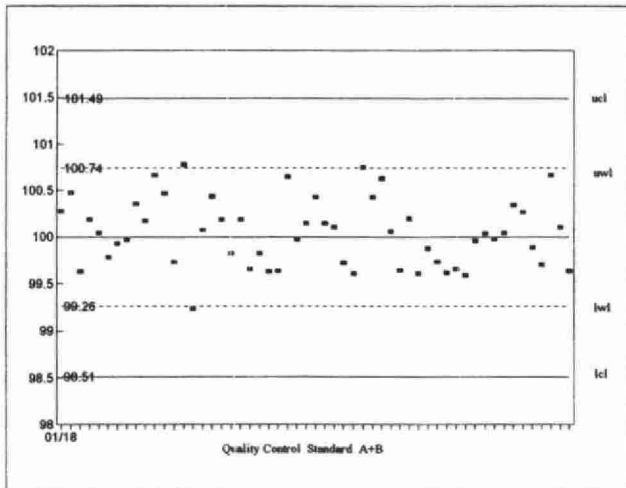
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
50	0.00 - 10.0	0.194	5.0
23	10.1 - 20.0	0.134	0.9
47	20.1 - 50.0	0.333	1.1
15	50.1 - 100	0.371	0.5
135	Overall	0.304	1.3

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	55	0.065	0.098

CHLORIDE (mg/L as Cl)
QUALITY CONTROL DATA FROM 01/01/99 TO 31/12/99
E3016



CHLORIDE

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	01/04/78
Method Reference No.	E3147	Reporting Unit	mg/L as Cl
LIMS Product Code	ANION3147	Supervisor	F. Tomassini
Sample Type/Matrix	Precipitation, Leachate, Surface Water		

SAMPLING:

Quantity Required	15 mL
Container	Glass or Plastic

ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with $\text{Na}_2\text{CO}_3/\text{NaHCO}_3$ to match the eluent strength and maintain background conductivity. The concentration of chloride in mg/L as Cl is determined by the comparison of the sample peak heights to a series of standards.

Sulphate is determined simultaneously.

INSTRUMENTATION:

Modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing, and partial data processing.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.01	Current T value: 0.05
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 2 standards, e.g., QCA
Drift	1 standard every 10 samples.

CHLORIDE (E3147)

QUALITY CONTROL DATA FROM 14/01/99 TO 15/12/99

Full Scale: to 2.0 mg/L as Cl

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	25	1.60	1.623	-0.023	0.0184
B:	25	0.40	0.412	-0.012	0.0121
A+B:		2.00	2.035	-0.035	0.0279
A-B:		1.20	1.212	-0.012	0.0141

s.d.(AB) S(between runs): 0.0156 Sw(within run): 0.0099 S/Sw: 1.57

The calibration is accepted if the calibration control values obtained lie within the ranges:

1.92 - 2.08 for A+B
1.14 - 1.26 for A-B

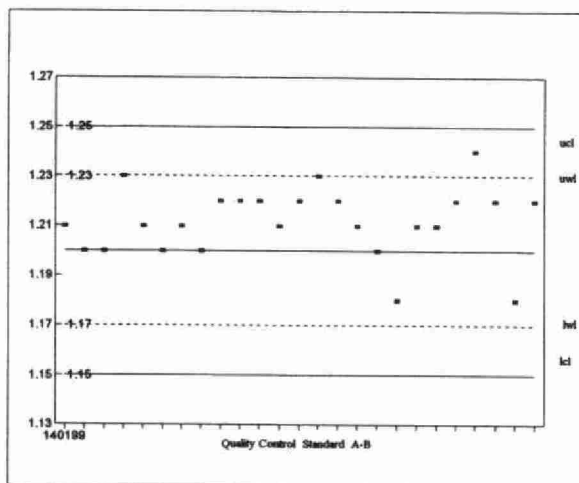
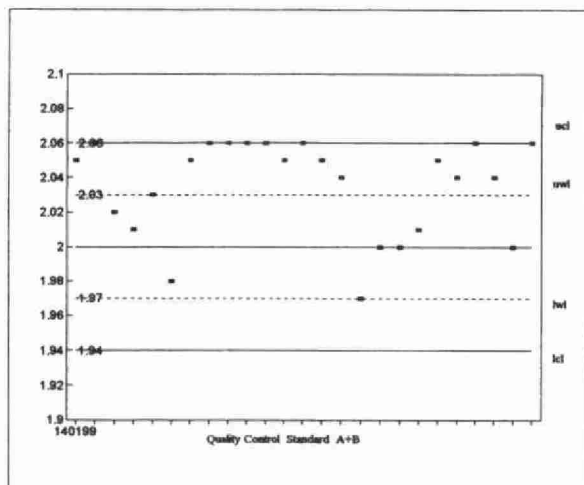
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
19	0.10 - 0.20	0.0117	10.7
26	0.21 - 0.40	0.0166	5.7
24	0.41 - 1.00	0.0313	5.0
13	1.01 - 2.00	0.0279	2.0
82	Overall	0.0230	5.4

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	25	-0.0072	0.0106

CHLORIDE (mg/L as Cl)
QUALITY CONTROL DATA FROM 14/01/99 TO 15/12/99
E3147



CHLORIDE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No	E3148	Reporting Unit	µg/Filter as Cl
LIMS Product Code	LOV3148, ANLOV3148	Supervisor	P. Wilson
Sample Type/Matrix	Air, Sequential and LoVol filters		

SAMPLING:

Quantity Required	1 filter
Container	50 mL polypropylene tube

SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of Pure-DW in polypropylene tubes with ultrasonic treatment followed by a 24 hour rest period.

ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of chloride in mg/L as Cl is determined by the comparison of the sample peak heights to a series of standards. Results are converted to µg/filter as Cl.

Nitrogen-nitrate and sulphate are determined simultaneously.

INSTRUMENTATION:

Ultrasonic bath; modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing and partial data processing.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.02 mg/L	Current T value: 0.10 mg/L
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CALIBRATION:

BL plus 9 standards

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
Drift	1 standard every 10 samples

NOTES:

To convert unit from mg/L to µg/Filter, the concentration of Cl in mg/L is multiplied by 50 for the W40 filters.

CHLORIDE (E3148)

QUALITY CONTROL DATA FROM 26/01/99 TO 06/12/99

Full Scale: to 2.0 mg/L as Cl

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	11	1.60	1.602	0.002	0.0060
B:	11	0.40	0.390	-0.010	0.0158
A+B:		2.00	1.991	-0.009	0.0170
A-B:		1.20	1.213	0.013	0.0168

s.d.(AB) S(between runs): 0.0135 Sw(within run): 0.0133 S/Sw: 1.0

The calibration is accepted if the calibration control values obtained lie within the ranges:

1.93 - 2.07 for A+B
1.15 - 1.25 for A-B

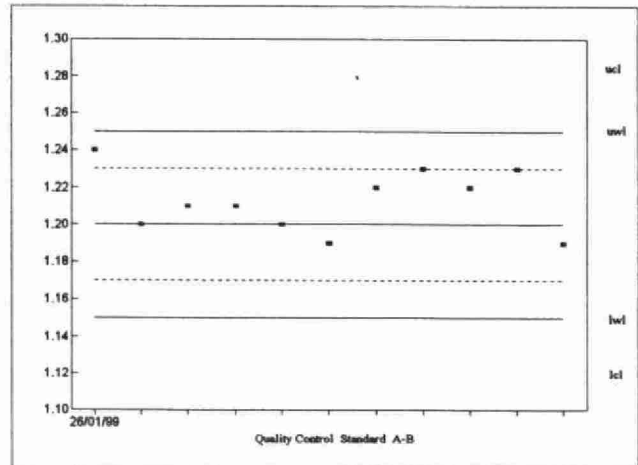
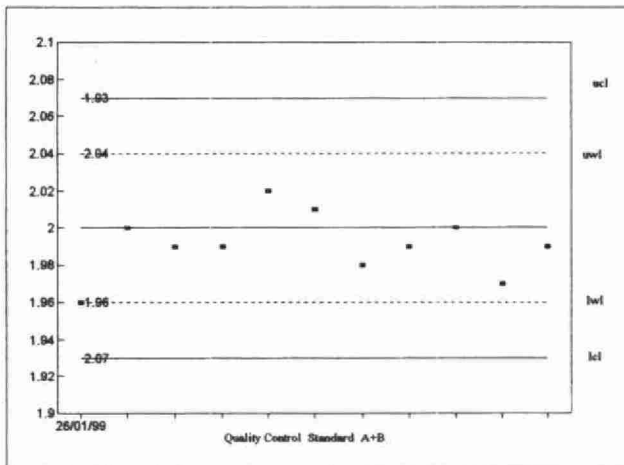
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
2	0 - 1.00	0.0127	2.0
24	1.01 - 2.00	0.0091	0.9
27	Overall	0.0124	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	11	0.0182	0.0603

CHLORIDE (mg/L as Cl)
QUALITY CONTROL DATA FROM 26/01/99 TO 06/12/99
E3148



CHLORIDE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No	E3372	Reporting Unit	mg/L as Cl
LIMS Product Code	ANION3372	Supervisor	P. Wilson
Sample Type/Matrix	Precipitation		

SAMPLING:

Quantity Required	15 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with $\text{Na}_2\text{CO}_3/\text{NaHCO}_3$ to match the eluent strength and maintain background conductivity. The concentration of chloride in mg/L as Cl is determined by the comparison of the sample peak heights to a series of standards.

Nitrogen-nitrate and sulphate are determined simultaneously.

INSTRUMENTATION:

Modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing, and partial data processing.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.01	Current T value: 0.05
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
Drift	1 standard every 10 samples

CHLORIDE (E3372)

QUALITY CONTROL DATA FROM 19/01/99 TO 23/12/99

Full Scale: to 1.0 mg/L as Cl

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	19	0.80	0.802	0.002	0.0170
B:	19	0.20	0.202	0.002	0.0155
A+B:		1.00	1.004	0.004	0.0201
A-B:		0.60	0.6004	0.0004	0.0256

s.d.(AB) S(between runs): 0.016 Sw(within run): 0.018 S/Sw: 0.9

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.96 - 1.04 for A+B
0.57 - 0.63 for A-B

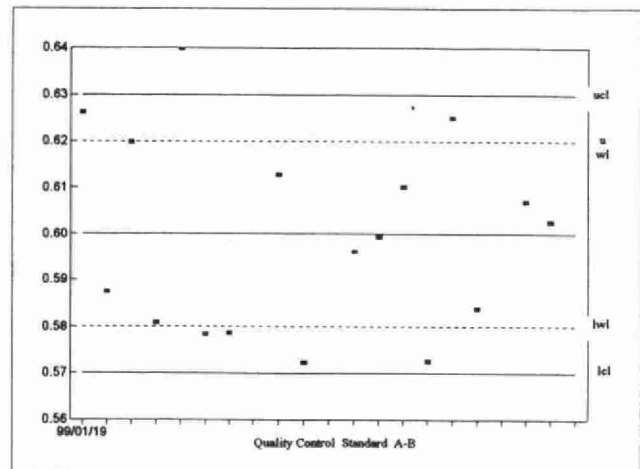
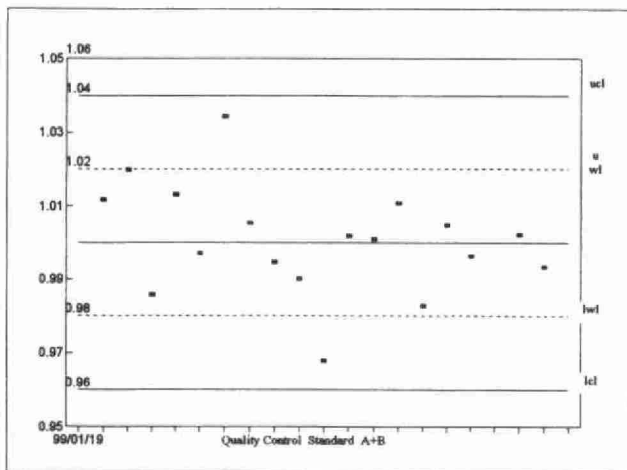
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
21	0 - 0.20	0.0078	11.5
15	0.21 - 0.50	0.0066	1.8
5	0.51 - 1.00	0.0048	0.5
41	Overall	0.0071	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	19	0.004	0.010

CHLORIDE (mg/L as Cl)
QUALITY CONTROL DATA FROM 19/01/99 TO 23/12/99
E3372



CHLORINE, TOTAL RESIDUAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	08/03/93
Method Reference No.	E3309	Reporting Unit	µg/L as Cl ₂
LIMS Product Code	RCL3309	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Raw Sewage, Surface Water, Drinking Water		

SAMPLING:

Quantity Required:	1 L
Container:	Narrow neck low actinic glass

ANALYTICAL PROCEDURE:

Samples are analyzed by amperometric titration. The sample pH is adjusted to between 3.5 and 4.5 with acetate buffer and excess KI is added.

INSTRUMENTATION:

Autoburette

REPORTING:

Maximum Significant Figures: 3	Current W value: 2	Current T value: 5
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CALIBRATION:

None

CONTROLS:

Performance Check	BL plus 2 QC standards, e.g., QCA
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NOTES:

No data available for 1999.

CHLOROPHYLL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/75
Method Reference No	E3169	Reporting Unit	µg/L
LIMS Product Code	CHL3169	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Drinking Water, Surface Water		

SAMPLING:

Quantity Required	1000 mL for clear samples; 500 mL if visibly green
Container	Glass or plastic
Other	In the field a sample is filtered through a nylon filter. The filter is folded and then placed between two membrane filter-support pads, and the package is enclosed in a plastic dish labelled with the sample number and sample volume filtered, the dish is kept in the dark or wrapped in aluminum foil, and shipped immediately, or kept frozen.

ANALYTICAL PROCEDURE:

Using a Commodore PET microcomputer-controlled, automated spectrophotometer, two scans are developed with absorbance measurements at 630, 645, and 663 nm for the first scans; the minimum absorbance value between 710 and 750 nm (readings at 5 nm intervals) is utilized as a turbidity correction. Chlorophyll "a" and "b" are calculated from this scan. After automated acidification, the second scan is obtained from the wavelength 665 nm for correcting chlorophyll "a" measurement. SCOR-UNESCO equations are used for all chlorophyll calculations.

INSTRUMENTATION:

- Automated modular continuous flow scanning spectrophotometer system
- Microcomputer system for control of sampling, timing and data processing (i.e. data capture, calculations and transfer of results to LIMS)

REPORTING:

Chlorophyll a; corrected Chlorophyll a; total Chlorophyll b; total	Maximum Significant Figures: 3	Current W value: 1.0 Current W value: 0.2 Current W value: 0.1	Current T value: 5.0 Current T value: 1.0 Current T value: 0.5
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CONTROLS:

Calibration	LTBL plus 2 "standards", e.g.QCA
Drift	"standard", BL every 20 samples

NOTES:

"Standards" are prepared from chlorophyll "a" and "b", but the materials are neither analytical grade nor are their solutions stable. Thus calibration controls are based on measured averages.

CHLOROPHYLL "a" (E3169)

QUALITY CONTROL DATA FROM 07/01/99 TO 08/12/99

Reporting Unit: µg/L

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	27	3.0	2.91	-0.09	0.1087
B:	27	1.0	0.998	-0.002	0.0710
A+B:		4.0	3.91	-0.09	0.1617
A-B:		2.0	1.91	-0.09	0.0870

s.d.(AB) S(between runs): 0.09 Sw(within run): 0.06 S/Sw: 1.5

The calibration is accepted if the calibration control values obtained lie within the ranges:

3.6 - 4.4 for A+B
1.7 - 2.3 for A-B

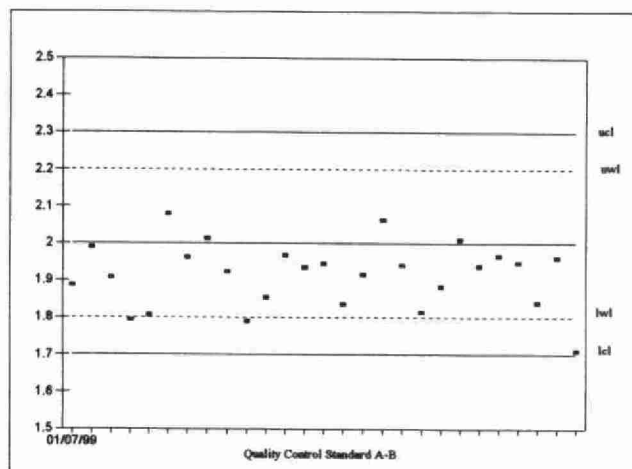
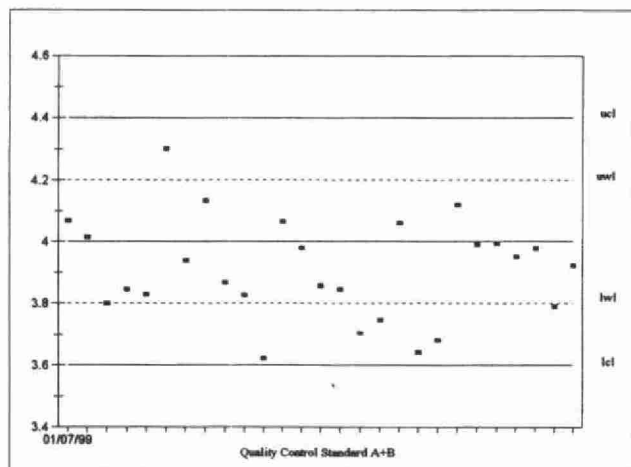
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
38	0 - 5.0	0.2548	22.6
1	5.1 - 10.0	N.A	N.A.
1	10.1 - 25.0	N.A	N.A.
40	Overall	0.2569	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	27	0.1099	0.0391
Filtered Blank	27	0.1443	0.0593

CHLOROPHYLL "a" (ug/L)
QUALITY CONTROL DATA FROM 07/01/99 TO 08/12/99
E3169



CHLOROPHYLL "a", ACIDIFIED (E3169)

QUALITY CONTROL DATA FROM 07/01/99 TO 08/12/99

Reporting Unit: µg/L

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	27	2.4	2.52	0.12	0.1764
B:	27	0.8	0.81	0.01	0.1336
A+B:		3.2	3.33	0.13	0.2587
A-B:		1.6	1.71	0.11	0.1816

s.d.(AB) S(between runs): .16 Sw(within run): 0.13 S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

2.4 - 4.0 for A+B
1.0 - 2.2 for A-B

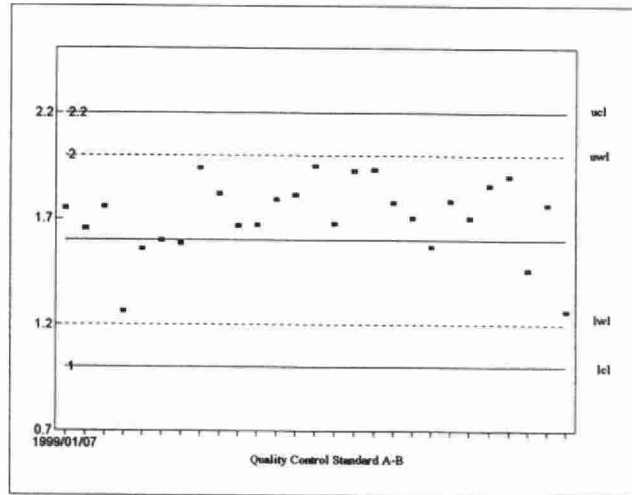
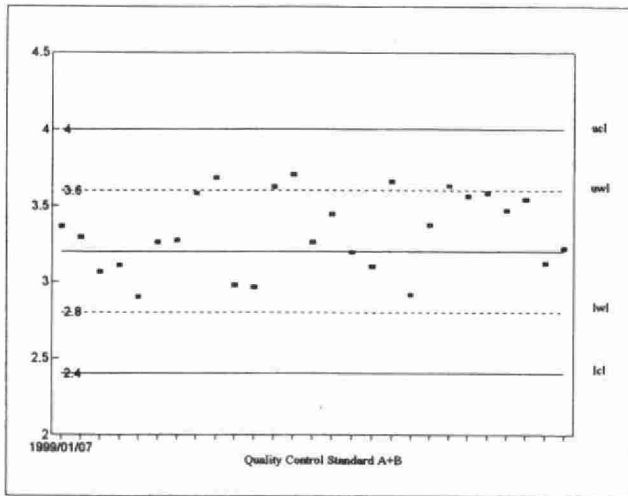
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
30	-0.5 - 1.0	0.3240	98.3
6	1.1 - 2.0	0.2170	16.9
2	2.1 - 5.0	N.A.	N.A.
0	5.1 - 10.0	N.A.	N.A.
1	10.1 - 100	N.A.	N.A.
39	Overall	0.3135	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	27	0.0364	0.1045
Filtered Blank	27	0.0288	0.1724

CHLOROPHYLL "a" , ACIDIFIED (µg/L)
 QUALITY CONTROL DATA FROM 07/01/99 TO 08/12/99
 E3169



CHLOROPHYLL "b" (E3169)

QUALITY CONTROL DATA FROM 07/01/99 TO 08/12/99

Reporting Unit: µg/L

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	27	3.0	3.1	0.1	0.1007
B:	27	1.0	1.1	0.1	0.0637
A+B:		4.0	4.2	0.2	0.1385
A-B:		2.0	1.98	-0.02	0.0959

s.d.(AB) S(between runs):0.08 Sw(within run): 0.07 S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

3.6 - 4.4 for A+B
1.7 - 2.3 for A-B

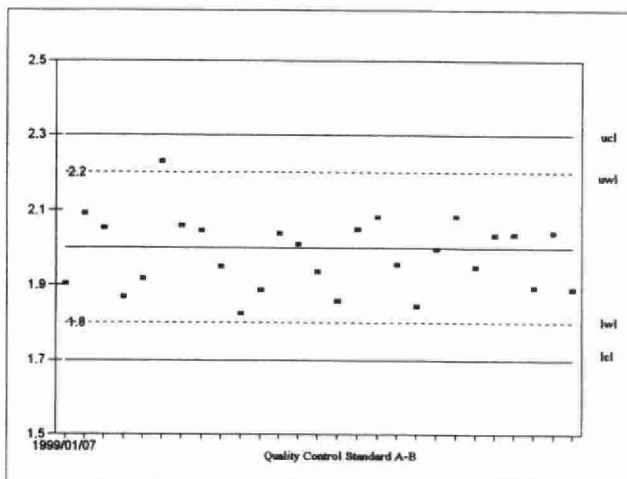
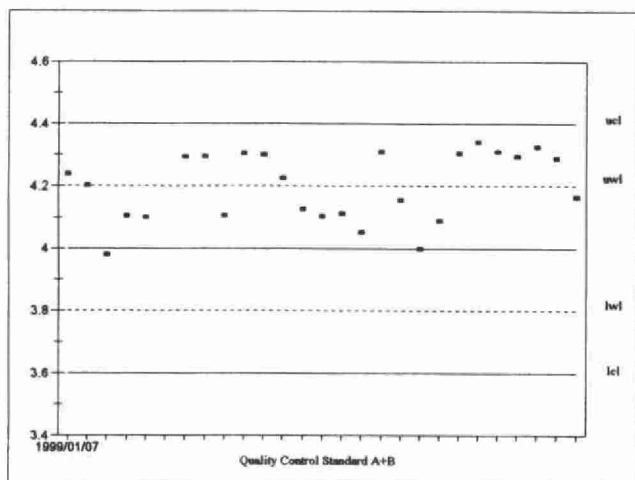
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
32	0 - 1.0	0.1646	294
7	1.1 - 2.0	0.1567	13.
1	2.1 - 5.0	N.A.	N.A.
40	Overall	0.1612	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	27	0.1569	0.0639
Filtered Blank	27	0.1959	0.0768

CHLOROPHYLL "b" ($\mu\text{g/L}$)
 QUALITY CONTROL DATA FROM 07/01/99 TO 08/12/99
 E3169



COLOUR, TRUE

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	15/10/80
Method Reference No.	E3025	Reporting Unit	TCU
LIMS Product Code	COL3025	Manager	F. Tomassini
Sample Type/Matrix	Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required:	25 mL
Container:	Glass or plastic

ANALYTICAL PROCEDURE:

True colour is measured on a settled sample colourimetrically in a system calibrated with acidified chloroplatinate standards. Colour is measured using a 400-450 nm broadband blue filter.
Approximate absorbance: 0.20 at the full scale level.

INSTRUMENTATION:

One colourimeter with broadband blue filter (400-450 nm)
One autosampler and chart-recorder
One Gilson pump

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1
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CALIBRATION:

7 acidified chloroplatinate standards, 2.5, 10, 20, 40, 60, 80, 100 TCU

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA, QCB, QCC
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NOTES:

Method was discontinued in Feb 1999. Analysis for colour was transferred to method E3219 under LIMS product code COL3219.

COLOUR, TRUE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	13/03/84
Method Reference No.	E3219	Reporting Unit	TCU
LIMS Product Code	COL3219	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Drinking Water, Ground Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

True colour is measured colourimetrically on the supernatant of a settled sample in a system calibrated with acidified chloroplatinate standards. The sample stream is measured using a broadband blue filter. Residual turbidity effects are suppressed by using a broadband red filter and increased path length in the reference stream.

Approximate absorbance: 0.3 at the full scale level.

INSTRUMENTATION:

Basic automated modular continuous flow system. Colour measurement is through a 3.0 cm. light path using a broadband filter (400-450 nm). Turbidity measurement is through a 5.0 cm. light path using a different broadband filter (660-740 nm). Data capture, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1
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CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
Drift	BL and standard after every 10 samples

NOTES:

The HP data capture/processing system was replaced by Labtronics in November 1998.

COLOUR, TRUE (E3219)

QUALITY CONTROL DATA FROM 12/02/99 TO 23/12/99

Laboratory Unit: Colourimetry Analytical Range: to 100 TCU

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	44	70.0	70.33	0.33	0.585
B:	44	25.0	25.56	0.56	0.417
C:	44	7.5	7.34	-0.16	0.310
A+B:		95.0	95.89	0.89	0.725
A-B:		45.0	44.77	-0.23	0.713
B+C:		32.5	32.90	0.40	0.645
B-C:		17.5	18.22	0.72	0.352

s.d.(AB) S(between runs): 0.51

s.d.(BC) S(between runs): 0.37

Sw(within run): 0.50

Sw(within run): 0.25

S/Sw: 1.01

S/Sw: 1.48

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

92.2	-	97.8	for	A+B
42.9	-	47.1	for	A-B
30.6	-	34.3	for	B+C
16.1	-	18.9	for	B-C

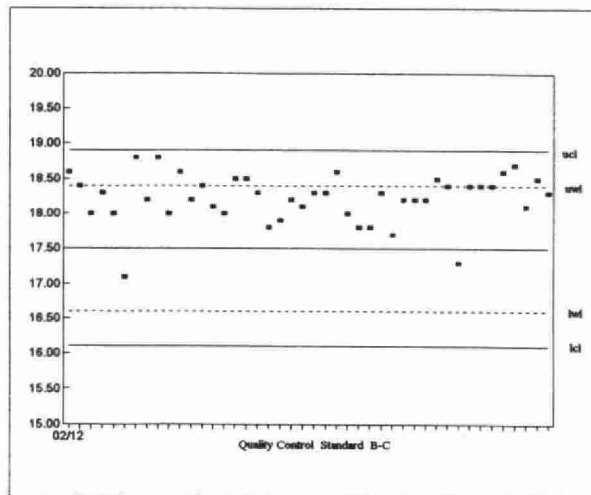
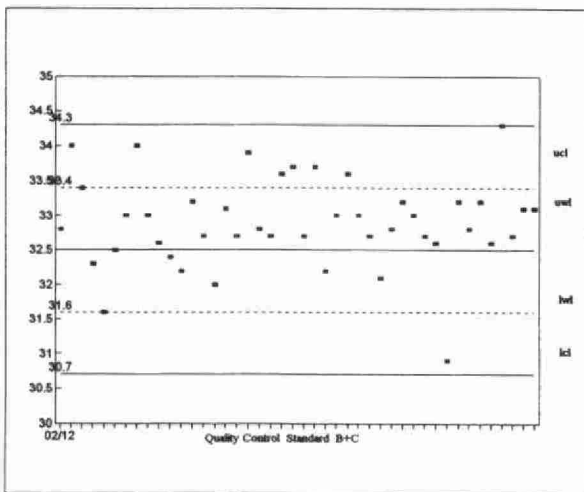
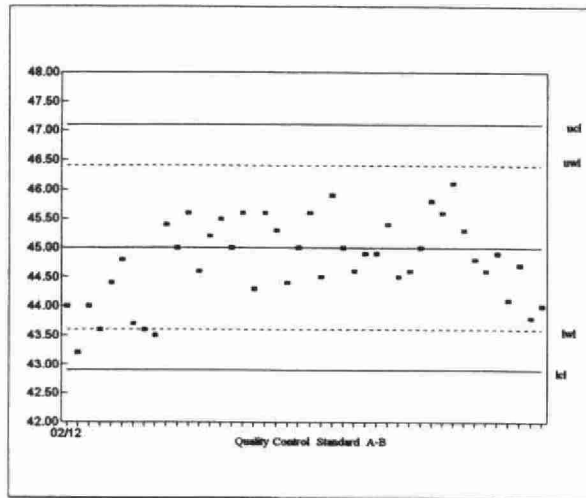
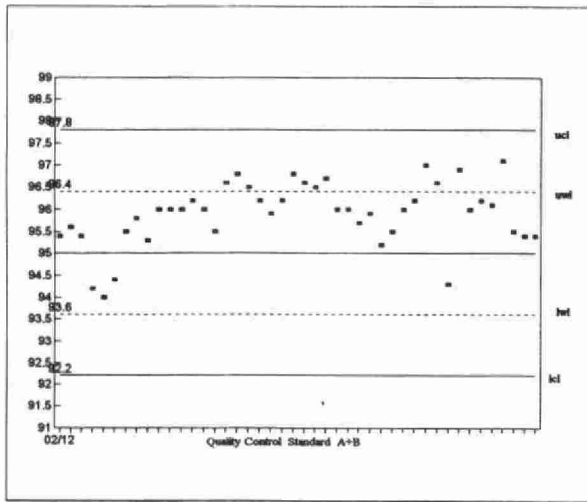
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
63	0 - 10.0	0.347	11.1
19	10.1 - 20.0	0.452	3.0
30	20.1 - 50.0	0.653	2.0
12	50.1 - 100.0	0.910	1.4
124	Overall	0.525	

OTHER CHECKS:

	n	Data Mean	Standard (1) Deviation
Long Term Blank	44	-0.459	0.286

COLOUR TRUE (TCU)
'QUALITY CONTROL DATA FROM 02/12/99 TO 23/12/99
E3219



CONDUCTIVITY

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	01/06/76
Method Reference No.	E3024	Reporting Unit	$\mu\text{S}/\text{cm}$ at 25°C
LIMS Product Code	COND3024	Supervisor	F. Tomassini
Sample Type/Matrix	Surface Water, Precipitation, Leachate		

SAMPLING:

Quantity Required	75 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

The sample is introduced into a jacketed conductivity cell. The conductivity is calculated from the chart record.

INSTRUMENTATION:

Conductivity meter with cell enclosed in a water jacket; temperature controlled water circulator. One autosampler, Gilson pump and dual-range chart recorder.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1
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CALIBRATION:

5 KCl standards, 10.2, 30.6, 50.8, 101.1, 151 μS

CONTROLS:

Calibration	LTBL plus 4 standards, e.g. QCA
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NOTES:

The control standards are corrected for the LTB from which they are made.

CONDUCTIVITY (E3024)

QUALITY CONTROL DATA FROM 11/01/99 TO 20/12/99

Analytical Range: to 500 $\mu\text{S}/\text{cm}$

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	23	146.7	147.8	1.05	1.2777
B:	23	51.8	52.3	0.48	1.0364
C:	23	51.8	52.8	1.00	0.7657
D:	23	14.9	15.0	0.13	0.7100
A+B:		198.5	200.0	1.5	1.5816
A-B:		94.9	95.5	0.57	1.7065
C+D:		66.7	67.8	1.13	0.9425
C-D:		36.9	37.7	0.77	0.7100

s.d.(AB) S(between runs): 1.16

s.d.(CD) S(between runs): 0.74

Sw(within run): 1.12

Sw(within run): 0.68

S/Sw: 1.04

S/Sw: 1.08

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

195	-	202	for	A+B
92.2	-	97.6	for	A-B
64.9	-	68.5	for	C+D
35.6	-	38.2	for	C-D

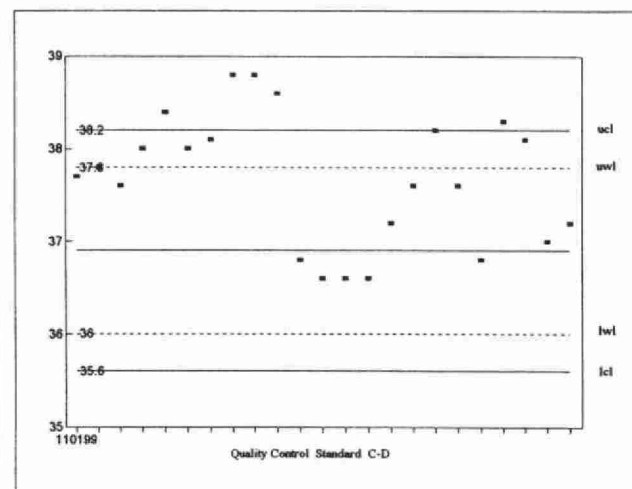
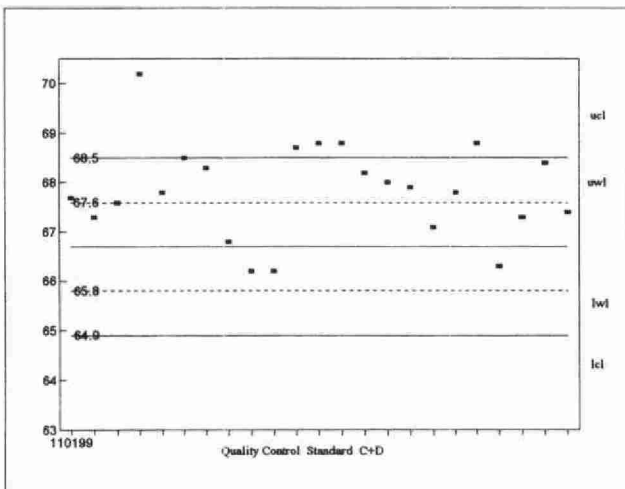
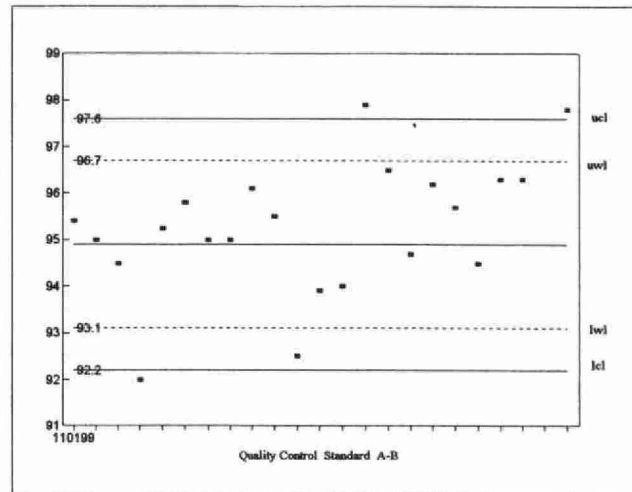
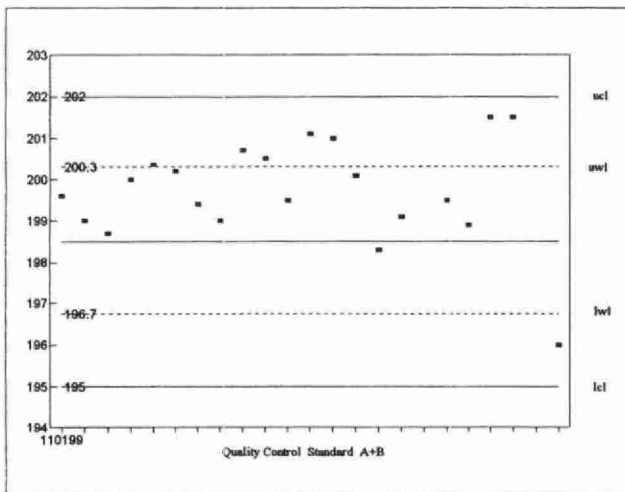
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
65	0 - 50.0	0.2659	1.03
13	50.1 - 100.0	0.5512	0.89
8	100.1 - 250.0	1.9521	1.3443
0	250.1 - 500.0	N.A.	N.A.
86	Overall	0.6737	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	23	-0.2135	1.3035

CONDUCTIVITY (us/cm)
QUALITY CONTROL DATA FROM 11/01/99 TO 20/12/99
E3024



CONDUCTIVITY

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced:	01/04/78
Method Reference No.	E3177	Reporting Unit:	µS/cm at 25°C
LIMS Product Code	COND3177	Supervisor:	P. Wilson
Sample Type/Matrix	Precipitation		

SAMPLING:

Quantity Required:	15 mL
Container:	Glass or plastic

ANALYTICAL PROCEDURE:

After equilibration at 25°C, The conductivity of the sample is measured.

INSTRUMENTATION:

Automated modular continuous flow conductivity system comprised of sampler, water bath, conductivity meter with cell, chart recorder.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1
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CALIBRATION:

1 standard

CONTROLS:

Calibration:	LTBL plus 2 standards, e.g. QCA
Drift:	1 solution every 10 samples

NOTES:

A calibration standard for the ion chromatographic system is used to monitor the drift for the conductivity system, but its theoretical conductivity is unknown.

CONDUCTIVITY (E3177)

QUALITY CONTROL DATA FROM 14/01/98 TO 26/04/00

Full Scale: to 100.0 $\mu\text{S/cm}$

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	34	44.5	45.02	-0.52	0.6509
B:	34	7.5	7.89	-0.39	0.4181
A+B:		52.0	52.92	-0.92	0.8147
A-B:		37.0	37.13	-0.13	0.7302

s.d.(AB)

S(between runs): 0.55

Sw(within run): 0.52

S/Sw: 1.06

The calibration is accepted if the calibration control values obtained lie within the ranges:

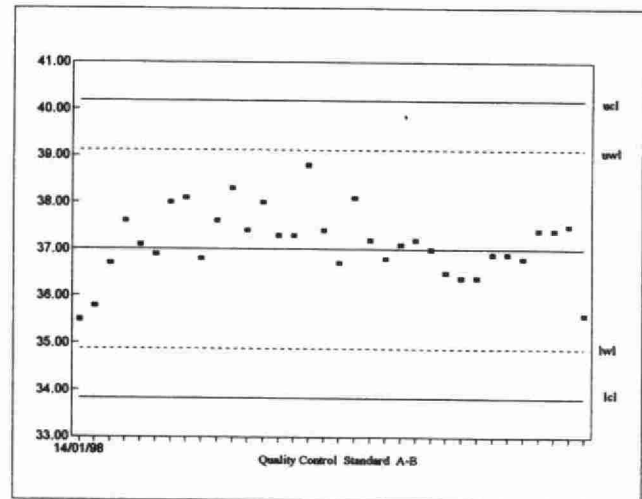
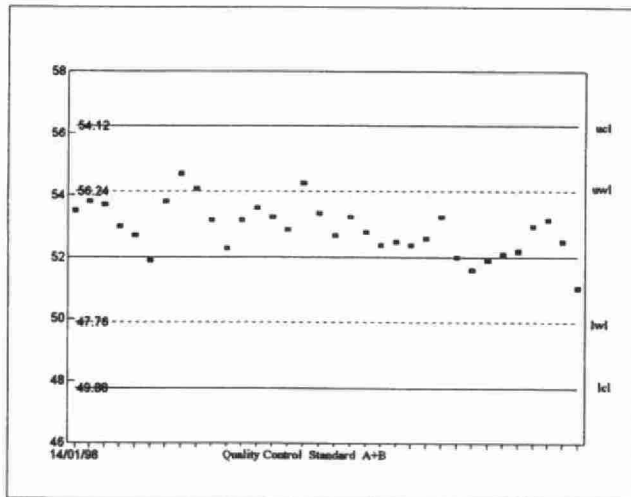
47.76 - 56.24 for A+B

33.82 - 40.18 for A-B

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
26	0 - 20.0	0.2707	2.3
38	20.1 - 50.0	0.6401	2.0
7	50.1 - 100.0	0.6325	1.0
71	Overall	0.5344	

CONDUCTIVITY ($\mu\text{S}/\text{cm}$)
QUALITY CONTROL DATA FROM 14/01/98 TO 26/04/00
E3177



CONDUCTIVITY

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced:	01/04/74
Method Reference No:	E3218	Reporting Units:	$\mu\text{S/cm}$ at 25°C
LIMS Product Code:	PHALCO3218,CONDPH3218	Supervisor:	P. Wilson
Sample Type/Matrix:	Sludge, Effluent, Industrial Waste, Raw Sewage, Drinking Water, Ground Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required:	50 mL
Container:	Glass or plastic

ANALYTICAL PROCEDURE:

After equilibration at 25°C, the conductivity of the sample is measured. pH, and total fixed endpoint alkalinity are determined simultaneously.

INSTRUMENTATION:

Automated modular continual flow conductivity system comprising of a sampler and conductivity meter with cell plus microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 1	Current T value: 5
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CONTROLS:

Calibration:	LTBL plus 4 standards, e.g. QCA
Drift:	In run standards throughout the run (tap water diluted to 50% V/V)

CONDUCTIVITY (E3218)

QUALITY CONTROL DATA FROM 05/01/99 TO 29/12/99

Analytical Range: to 2000 $\mu\text{S}/\text{cm}$

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	112	1413	1410.3	-2.7	4.5332
B:	112	718	717.5	-0.5	2.7540
C:	112	147	148.2	1.2	0.7369
D:	112	37.1	38.3	1.2	0.4264
A+B:		2131	2127.8	-3.2	5.9871
A-B:		695	692.7	-2.3	4.5191
B+C:		865	865.7	0.7	3.0050
B-C:		571	569.3	-1.7	2.6879
C+D:		184.1	186.5	2.4	0.9494
C-D:		109.9	109.9	-0.0	0.7404

s.d.(AB) S(between runs): 3.75

Sw(within run): 3.20

S/Sw: 1.2

s.d.(BC) S(between runs): 2.02

Sw(within run): 1.90

S/Sw: 1.1

s.d.(CD) S(between runs): 0.60

Sw(within run): 0.52

S/Sw: 1.1

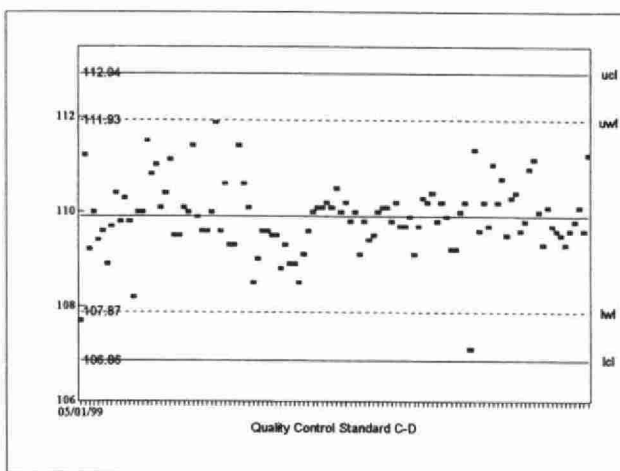
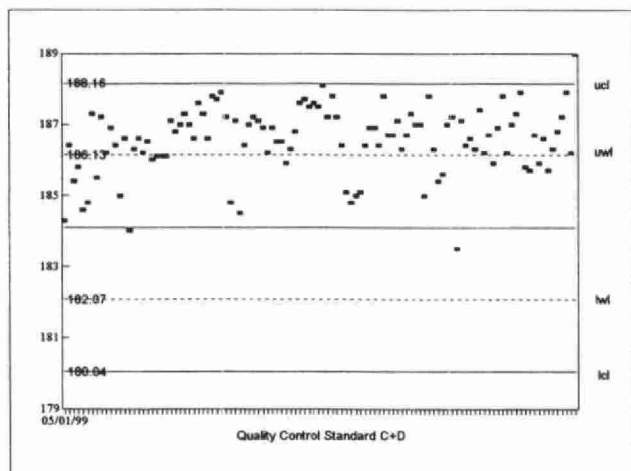
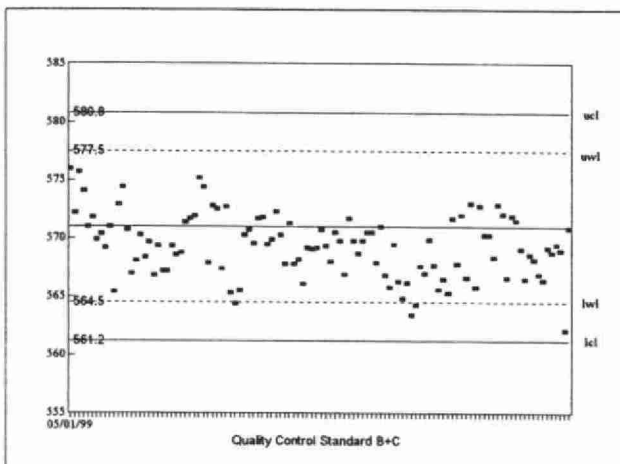
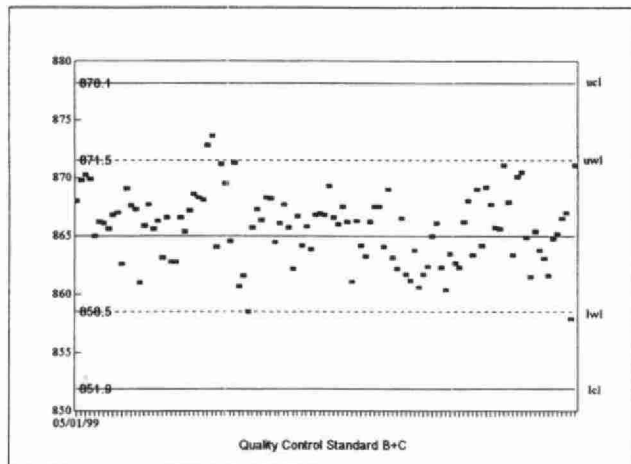
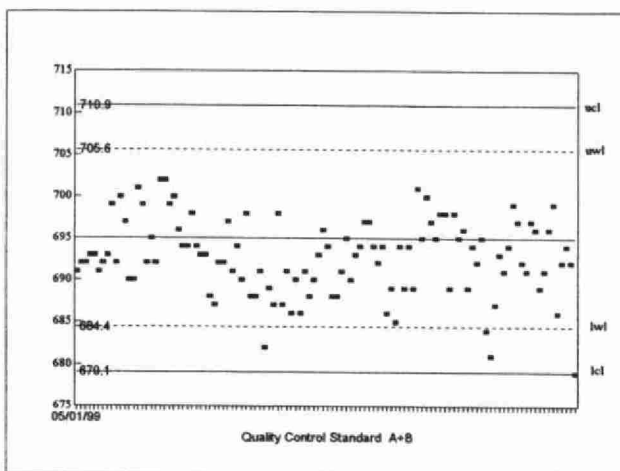
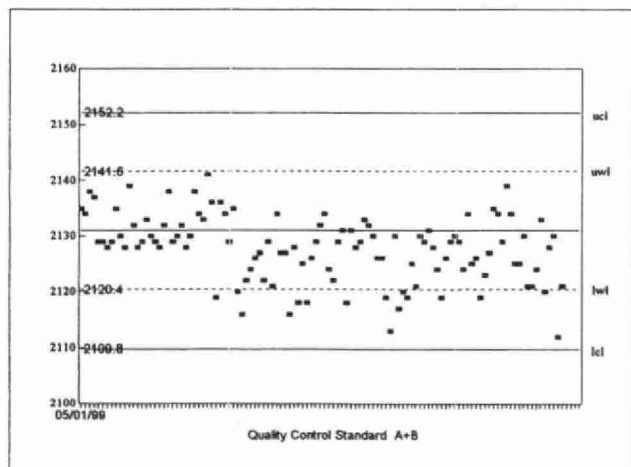
On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

2109.8	-	2152.8	for	A+B
679.1	-	711.7	for	A-B
851.9	-	876.64	for	B+C
561.2	-	580.8	for	B-C
180.04	-	188.16	for	C+D
106.86	-	579.68	for	C-D

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
173	0 - 400	1.8526	1.2
92	401 - 1000	3.1750	0.5
30	1001 - 2000	3.1919	0.2
15	2001 - 10000	7.6280	0.5
1	>10000	N.A.	N.A.
311	Overall	2.6017	

CONDUCTIVITY (uS/cm)
QUALITY CONTROL DATA FROM 05/01/99 TO 29/12/99
E3218



CYANIDE, FREE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/01/98
Method Reference No.	E3015	Reporting Unit	Aqueous: mg/L as CN^- Solid: $\mu\text{g/g}$ as CN^-
LIMS Product Code	CNF3015	Supervisor	P. Wilson
Sample Type/Matrix	Aqueous: Surface Water, Drinking Water, Ground Water, Raw Sewage & Effluent, Industrial Effluent. Solid: Soil, Sediment, Vegetation, Dried Sludge, Industrial Waste		

SAMPLING:

Quantity Required:	Aqueous: 500 mL + 10 drops of 50% w/v NaOH Solid : 5 g, minimum
Container:	Glass or plastic

ANALYTICAL PROCEDURE:

Free cyanides are the simple and weakly dissociable cyanides which form HCN upon acidification to pH4.0 (such as HCN and KCN). The automated determination of free cyanide exposes the sample to distillation which isolates HCN under specific acidic conditions. A zinc sulphate solution is included which eliminates interference from complexed iron cyanides. Cyanide is determined colourimetrically by the reaction of cyanide with chloramine -T to form cyanogen chloride which further reacts with a combination of barbituric acid and isonicotinic acid to form a highly coloured coupling product, which is measured at 600 nm.

Aqueous samples are introduced directly to the continuous flow system by an autosampler. Solid samples are extracted in a sodium hydroxide solution with mechanical shaking for 6 to 8 hours and then centrifuged. The supernatant is decanted, diluted if necessary to eliminate interference from colour and introduced to the continuous flow system by the autosampler. Solid samples are not dried or ground, but weighed and extracted as received, to prevent the loss of simple cyanides. If the sample is wet, results are reported as $\mu\text{g/g}$ wet and moisture content is reported by a separate method.

INSTRUMENTATION:

Skalar automated segmented flow colourimetric system, measurement through a 500 mm light path at 600 nm.

Skalar data capture and data processing software with computer system.

REPORTING:

Maximum Significant Figures: 3 or the nearest W	Current W value: 0.001 mg/L 0.01 $\mu\text{g/g}$	Current T value: 0.005mg/L 0.05 $\mu\text{g/g}$
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CALIBRATION:

BL plus 6 standards (S0 to S5)

CONTROLS:

Calibration:	LTB plus 2 standards , e.g. QCA
Drift:	BL and check standards

CYANIDE, FREE (E3015)

QUALITY CONTROL DATA FROM 05/01/99 TO 24/11/99

Laboratory Unit: Colourimetry

Full Scale: to 0.2 mg/L as CN

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	19	0.15	0.1503	0.0003	0.0020
B:	19	0.02	0.0187	-0.0013	0.0010
A+B:		0.17	0.1690	-0.0010	0.0020
A-B:		0.13	0.1316	0.0016	0.0024

s.d.(AB)

S(between runs): 0.0017

Sw(within run): 0.0016

S/Sw: 0.9

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.154 - 0.186 for A+B

0.118 - 0.142 for A-B

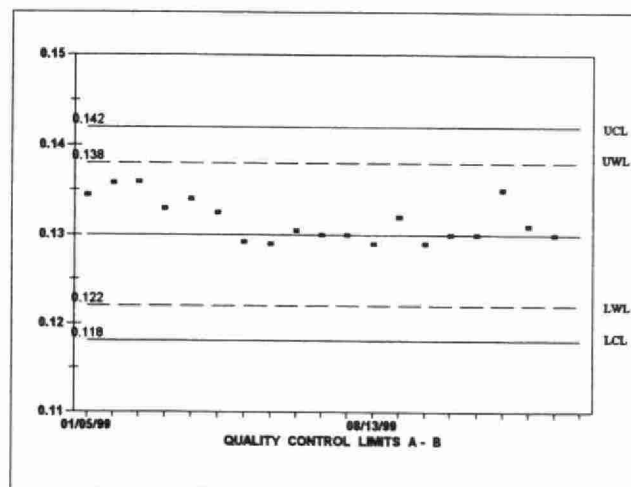
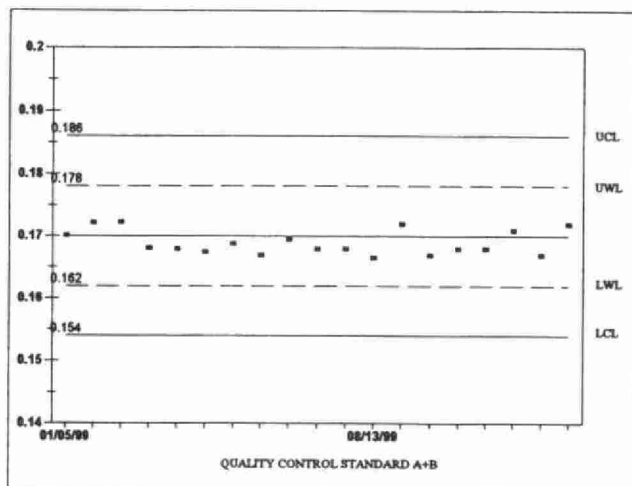
REFERENCE MATERIAL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
KCN:	19	0.10	0.0994	0.0006	0.0024
FeCN:	19	0.10	0.0054	0.0054	0.0037

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
40	0 - 0.020	0.0004	13.5
0	0.021 - 0.040	N.A.	N.A.
4	0.041 - 0.100	0.0014	0.9
0	0.101 - 0.200	N.A.	N.A.
44	Overall	0.0005	

CYANIDE FREE (mg/L as CN)
QUALITY CONTROL DATA FROM 05/01/99 TO 24/11/99
E3015



CYANIDE, TOTAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/01/98
Method Reference No.	E3015	Reporting Unit	Aqueous: mg/L as CN^- Solid: $\mu\text{g/g}$ as CN^-
LIMS Product Code	CN3015	Supervisor	P. Wilson
Sample Type/Matrix	Aqueous: Surface Water, Drinking Water, Ground Water, Raw Sewage & Effluent, Industrial Effluent. Solid: Soil, Sediment, Vegetation, Dried Sludge, Industrial Waste		

SAMPLING:

Quantity Required:	Aqueous: 500 mL + 10 drops of 50% w/v NaOH Solid : 5 g, minimum
Container:	Glass or plastic

ANALYTICAL PROCEDURE:

Total cyanides include free, simple (HCN , KCN) and weakly dissociable cyanides ($\text{Ni}(\text{CN})_4$) as well as those complexed cyanides that decompose to form free cyanides that distill out as HCN in an acidic environment. The automated determination of total cyanide exposes the sample to ultraviolet radiation to break down organic metallic and alkali-complexed cyanide compounds to free cyanide. The distillation step isolates HCN under specific acidic conditions. The sequential combination of UV digestion plus distillation yields the measurement of "total cyanide". Cyanide is measured colourimetrically by the reaction of cyanide with chloramine -T to form cyanogen chloride which further reacts with a combination of barbituric acid and isonicotinic acid to form a highly coloured coupling product, which is measured at 600 nm.

Aqueous samples are introduced directly to the continuous flow system from an autosampler. Solid samples are extracted in a sodium hydroxide solution with mechanical shaking for 6 to 8 hours, then centrifuged. The supernatant is then decanted, diluted if necessary to eliminate interference from colour and introduced to the continuous flow system by the autosampler. Solid samples are not dried or ground, but weighed and extracted as received, to prevent the loss of simple cyanides. If the sample is wet, results are reported as $\mu\text{g/g}$ wet and moisture content is reported by a separate method.

INSTRUMENTATION:

Skalar automated segmented flow colourimetric system, measurement through a 500 mm light path at 600 nm. Skalar data capture and data processing software with computer system.

REPORTING:

Maximum Significant Figures: 3 or the nearest W	Current W value: 0.001 mg/L 0.01 $\mu\text{g/g}$	Current T value: 0.005mg/L 0.05 $\mu\text{g/g}$
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CALIBRATION:

BL plus 6 standards (S0 to S5)

CONTROLS:

Calibration:	LTB plus 2 standards , e.g. QCA
Drift:	BL and check standards

CYANIDE, TOTAL (E3015)

QUALITY CONTROL DATA FROM 05/01/99 TO 09/12/99

Laboratory Unit: Colourimetry

Full Scale: to 0.2 mg/L as CN

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	28	0.15	0.15003	0.0003	0.0018
B:	28	0.02	0.01909	-0.0009	0.0010
A+B:		0.17	0.16910	-0.0010	0.0020
A-B:		0.13	0.13509	0.0009	0.0021

s.d.(AB)

S(between runs): 0.0015

Sw(within run): 0.0015

S/Sw: 1.0

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.154 - 0.186 for A+B

0.118 - 0.142 for A-B

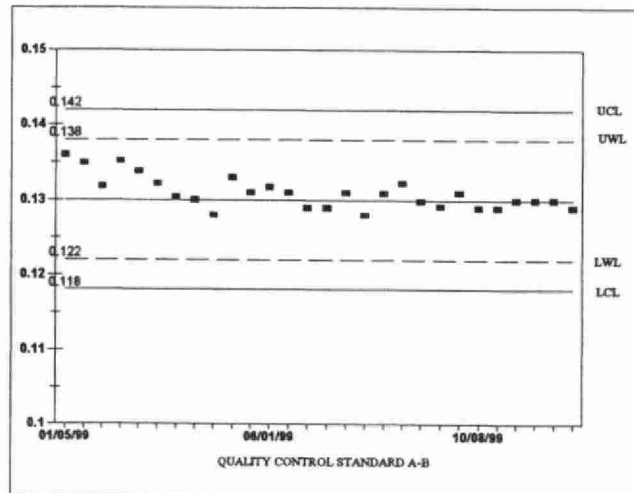
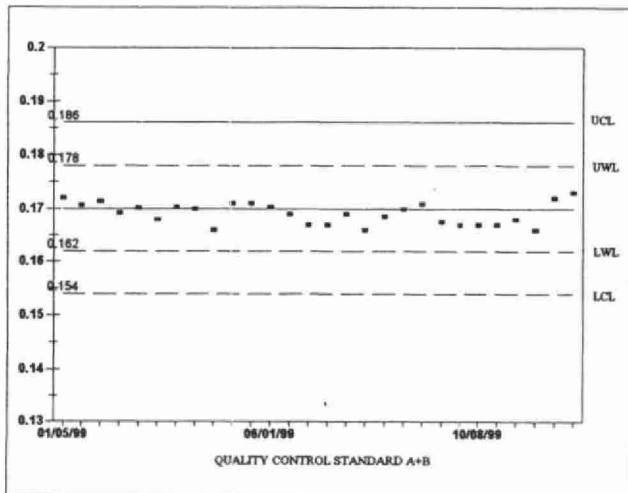
REFERENCE MATERIAL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
KCN:	28	0.10	0.097	-0.0029	0.0166
FeCN:	28	0.10	0.103	0.0032	0.0030

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
60	0 - 0.020	0.0004	12.2
0	0.021 - 0.040	N.A.	N.A.
6	0.041 - 0.100	0.0032	4.5
15	0.101 - 0.200	0.0018	1.3
81	Overall	0.0012	

CYANIDE TOTAL (mg/L as CN)
QUALITY CONTROL DATA FROM 05/01/99 TO 09/12/99
E3015



FLUORIDE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	before '74
Method Reference No.	E3369	Reporting Unit	mg/L as F
LIMS Product Code	F3369	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Process Water, Drinking Water, Ground Water, Surface Water, Leachate		

SAMPLING:

Quantity Required	50 mL
Container	Plastic

ANALYTICAL PROCEDURE:

Using an automated flow system the sample is distilled in the presence of sulphuric acid at 160°C; the distillate is then reacted (in an acetic acid-acetate buffer media) with Alizarin Fluorine Blue and lanthanum nitrate to form a ternary Alizarin Blue-lanthanide-fluoride complex.

Approximate absorbance: 0.8 at the full scale level.

INSTRUMENTATION:

Modular continuous flow colourimetric system plus a distillation module. Colourimetric measurement is through a 5.0 cm. light path at 630 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.01	Current T value: 0.05
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CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration	LTB plus 3 standards, e.g. QCA
Drift	BL every 10 samples; standard every 20 samples

NOTES:

The HP data capture/processing system was replaced by Labtronics in November 1999.

Feb 1998. The Nitrate plus Nitrite test was discontinued at E3369 and made available at E3364 and E3366. The LIMS product code changed from FNOT3369 to F3369.

FLUORIDE (E3369)

QUALITY CONTROL DATA FROM 05/01/99 TO 05/11/99

Laboratory Unit: Colourimetry

Full Scale: to 2.0 mg/L as F

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	31	1.6	1.6055	0.0055	0.0234
B:	31	0.8	0.7987	-0.0013	0.0171
C:	31	0.16	0.1597	-0.0003	0.0147
A+B:		2.4	2.4041	0.0041	0.0280
A-B:		0.8	0.8068	0.0068	0.0298
B+C:		0.96	0.9584	-0.0016	0.0266
B-C:		0.64	0.6390	-0.0010	0.0176

s.d.(AB) S(between runs):0.020

Sw(within run): 0.021

S/Sw: 0.97

s.d.(BC) S(between runs):0.016

Sw(within run): 0.012

S/Sw: 1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

2.302	-	2.498	for	A+B
0.726	-	0.874	for	A-B
0.91	-	1.01	for	B+C
0.59	-	0.69	for	B-C

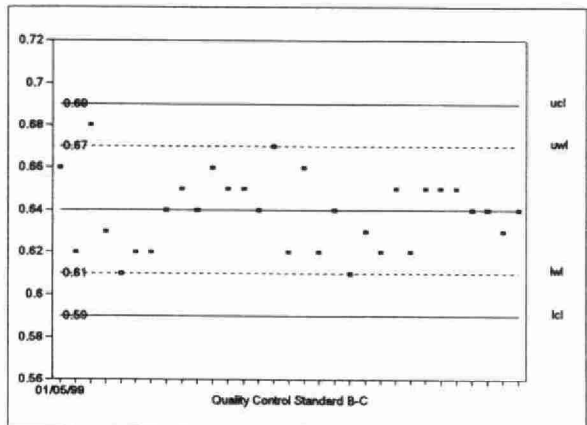
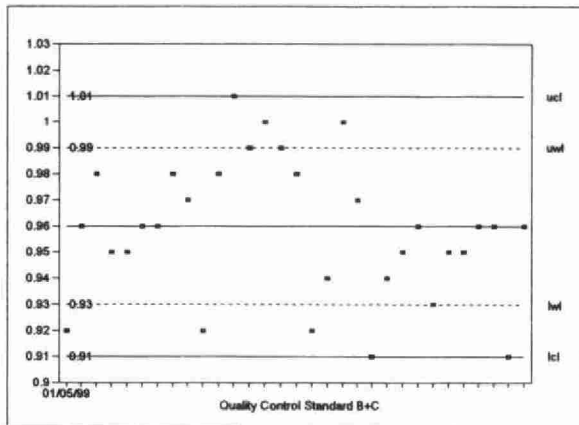
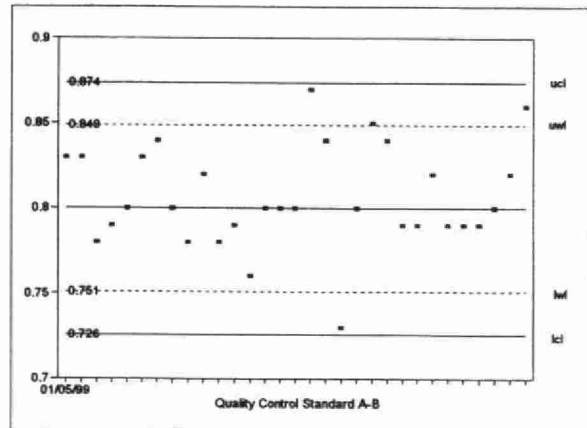
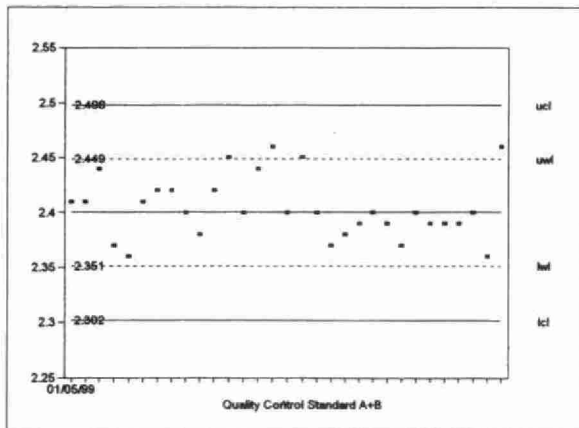
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
58	0 - 0.20	0.0075	7.1
8	0.21 - 0.40	0.0071	2.7
16	0.41 - 1.00	0.0106	1.4
9	1.01 - 2.00	0.0131	1.1
91	Overall	0.0088	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	31	-0.0013	0.0138

FLUORIDE (mg/L as F)
QUALITY CONTROL DATA FROM 05/01/99 TO 05/11/99
E3369



IRON, TOTAL

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	20/07/88
Method Reference No.	E3303	Reporting Unit	µg/L as Fe
LIMS Product Code	FEMN3303, FE3303	Manager	F. Tomassini
Sample Type/Matrix	Surface Water, Precipitation, Leachate		

SAMPLING:

Quantity Required	25 mL
Container	Glass or plastic, capped, acidified to 0.25% with HNO ₃

ANALYTICAL PROCEDURE:

An undigested sample is introduced to an in-line UV digester. A reducing agent and a buffer are added to the sample. TPTZ is added to develop a blue colour, the intensity of which is proportional to the concentration of Fe in the sample. The colour is measured at 600nm.

INSTRUMENTATION:

- An AAll autoanalyzer with colorimeter and automated sampler.

REPORTING:

Maximum Significant Figures: 3	Current W value: 2	Current T value: 10
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CALIBRATION:

BL plus 4 standards

CONTROLS:

Calibration	Long Term blank, 3 QC's, 4 duplicates
Drift	Blank plus 1 standard every 10 samples.

NOTES:

Method was discontinued in Feb 1999. Analysis for Iron was transferred to method E3386 under the LIMS product code MET3386.

MAGNESIUM

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	20/07/88
Method Reference No.	E3249	Reporting Unit	mg/L as Mg
LIMS Product Code	CAT3249	Supervisor	F. Tomassini
Sample Type/Matrix	Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	5 mL
Container	Plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 285.2 nm with an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level.

INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.005	Current T value: 0.025
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CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g., QCA
Drift	BL, reslope standard every 10 samples.

MAGNESIUM (E3249)

QUALITY CONTROL DATA FROM 12/01/99 TO 22/12/99

Full Scale: to 2.0 mg/L as Mg

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	47	1.6	1.615	0.016	0.0083
B:	47	0.4	0.401	0.001	0.0034
C:	47	0.1	0.103	0.003	0.0017
A+B:		2.0	2.017	0.017	0.0104
A-B:		1.2	1.214	0.014	0.0072
B+C:		0.5	0.504	0.004	0.0039
B-C:		0.3	0.299	-0.001	0.0036

s.d.(AB) S(between runs): 0.006 Sw(within run): 0.005 S/Sw: 1.24
s.d.(BC) S(between runs): 0.001 Sw(within run): 0.003 S/Sw: 0.48

The calibration is accepted if the calibration control values obtained lie within the ranges:

1.968 - 2.032 for A+B
1.176 - 1.224 for A-B
0.490 - 0.510 for B+C
0.293 - 0.307 for B-C

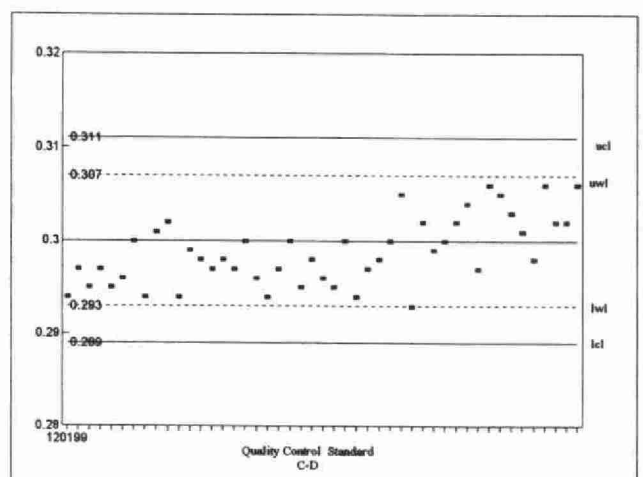
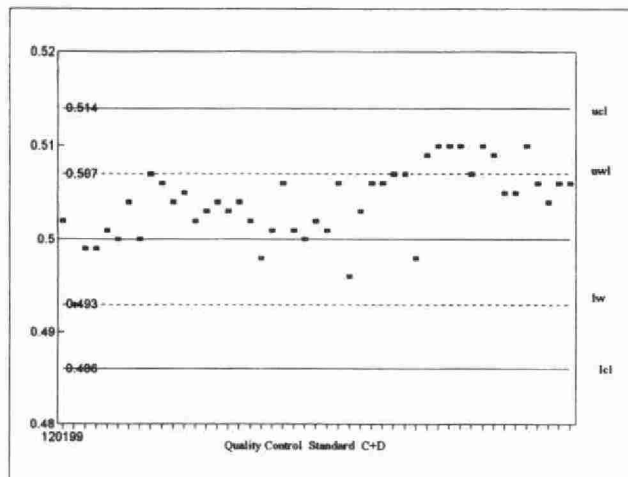
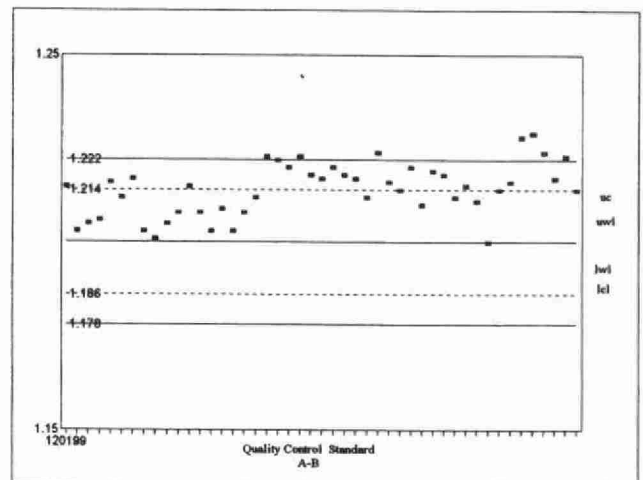
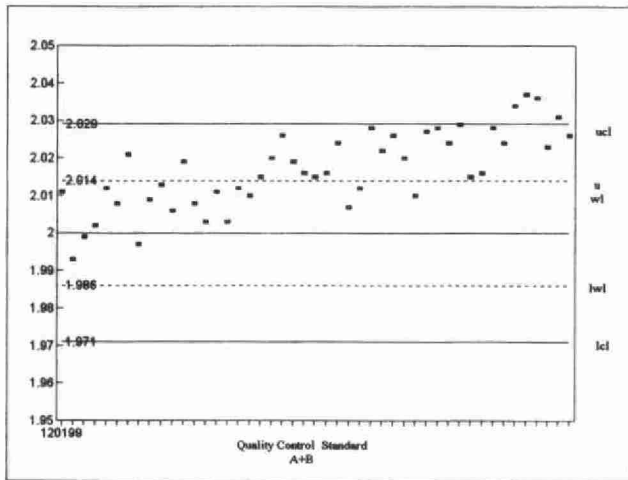
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
42	0.00 - 0.20	0.0016	2.7
48	0.21 - 0.40	0.0033	1.0
112	0.41 - 1.00	0.0352	5.1
20	1.01 - 2.00	0.0087	0.7
222	Overall	0.0252	4.6

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	47	-0.0003	0.0016

MAGNESIUM (mg/L as Mg)
QUALITY CONTROL DATA FROM 12/01/99 TO 22/12/99
E3249



MANGANESE, TOTAL

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	1991
Method Reference No.	E3303B	Reporting Unit	µg/L as Mn
LIMS Product Code	FEMN3303	Manager	F. Tomassini
Sample Type/Matrix	Surface Water, Precipitation, Leachate		

SAMPLING:

Quantity Required	25 mL
Container	Glass or plastic, capped, acidified to 0.25% with HNO ₃

ANALYTICAL PROCEDURE:

An undigested sample is introduced to an in-line UV digester. A reducing agent and an ammonium buffer are added to the sample. Formaldoxime complexes with Mn to develop a colour the intensity of which is proportional to the concentration of Mn in the sample. EDTA is then added to complex interferences. The color is read at 480nm. A reference channel is used to counter the effects of residual natural colour in the sample. In the reference channel the EDTA is added prior to the addition of colour reagent.

INSTRUMENTATION:

- An AAll autoanalyzer with colorimeter and automated sampler.

REPORTING:

Maximum Significant Figures: 3	Current W value: 1	Current T value: 5
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CALIBRATION:

BL plus 4 standards

CONTROLS:

Calibration	Long Term blank, 3 QC's, 4 duplicates
Drift	Blank plus 1 standard every 10 samples.

NOTES:

Method was discontinued in Feb 1999. Analysis for Manganese was transferred to method E3386 under the LIMS product code MET3386.

NITRATE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3004	Units	$\mu\text{g}/\text{m}^3$ as NO_3
LIMS Product Code	ANION3004	Supervisor	P. Wilson
Sample Type/Matrix	Air; HiVol Glass Fibre, Quartz and Polyflon, Other Filters and Puff		

SAMPLING:

Quantity Required	3/4" or 1.9cm strip from 8"x10" filter
Container	50 mL polypropylene tube

SAMPLING PREPARATION:

A 3/4" strip is cut in pieces and deposited into a 50 mL polypropylene tube. 50 mL of Pure-Water is added to the tube. The tube is placed on a horizontal shaker for approximately 1 hour. The supernatant is then filtered into a 15 mL plastic tube and analysed.

ANALYTICAL PROCEDURE:

Nitrate separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of sodium bicarbonate and sodium carbonate and a conductivity detector. The concentration of nitrate (mg/L) is determined by the comparison of the analyte peak area count to that of a series of standards. The analyte result is corrected for the filter blank before the final calculation is made. The result is reported as $\mu\text{g}/\text{m}^3$ as NO_3 .

Chloride and sulphate are determined simultaneously.

INSTRUMENTATION:

Horizontal Shaker, ion chromatographic system plus a PC with ChromPerfect software and DT2804 card for automated sample injection, timing, and data processing.

REPORTING:

Maximum Significant Figures: 2	Current W value: $0.1 \mu\text{g}/\text{m}^3$	Current T value: $0.5 \mu\text{g}/\text{m}^3$
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CALIBRATION:

6 standards

CONTROLS:

Calibration	MB, IS(n), CS1, and CS2
Drift	Duplicate plus 2 standards approximately every 20 samples

NITRATE cont'd

NOTES:

To convert unit from mg/L to $\mu\text{g}/\text{m}^3$, the final concentration of NO_3 in mg/L is multiplied by the following formula:

$$\text{Result (mg/L)} \times 50\text{mL} \times (63/6.75) / \text{air volume} = \mu\text{g}/\text{m}^3$$

Where: 63 is the area of the filter exposed and 6.75 is the sample aliquot area in square inch.

NITRATE (E3004)

QUALITY CONTROL DATA FROM 1997 TO 1999

Analytical Range: to 28.61 $\mu\text{g}/\text{m}^3$ as NO_3

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
172	0.00 - 2.86	0.080	12.2
20	2.89 - 7.15	0.097	2.2
5	7.18 - 14.31	0.089	0.8
0	14.33 - 24.90	N.A.	N.A.
197	Overall	0.082	

NITRILOTRIACETIC ACID

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	17/04/98
Method Reference No.	E3406	Reporting Unit	mg/L as NTA
LIMS Product Code	NTA3406	Supervisor	P. Wilson
Sample Type/Matrix	Drinking Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Nitriлотriacetate Acid is separated from other anions in the samples by automated suppressed gradient ion chromatography. A sodium hydroxide eluent is used with conductivity detection. The concentration of Nitriлотriacetate acid in mg/L as NTA is determined by comparison of the sample scan to a series of standard scans.

INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system with gradient flow control module .

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.01	Current T value: 0.05
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
Drift	1 standard every 10 samples

NITRILOTRIACETIC ACID (E3406)

QUALITY CONTROL DATA FROM 21/01/99 TO 23/12/99

Full Scale: to 1.00 mg/L as NTA

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	37	0.80	0.796	-0.004	0.0161
B:	37	0.20	0.194	-0.006	0.0134
A+B:		1.00	0.990	-0.010	0.0204
A-B:		0.60	0.602	0.002	0.0216

s.d.(AB) S(between runs): 0.0148 Sw(within run): 0.0153 S/Sw: 1.0

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.95 - 1.05 for A+B
0.56 - 0.64 for A-B

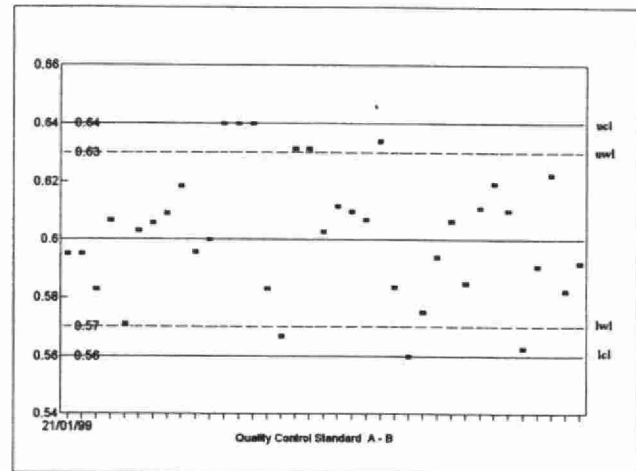
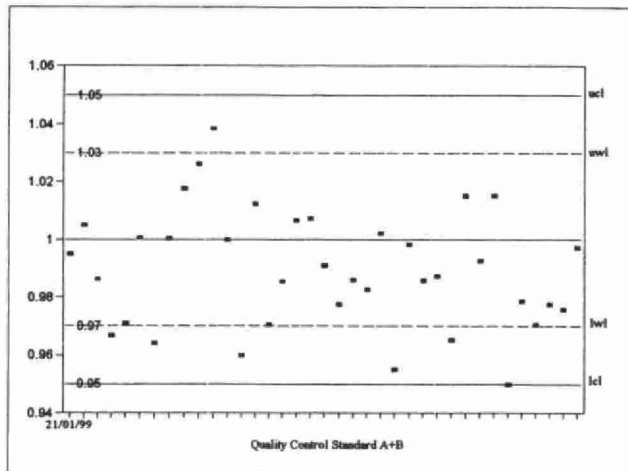
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
111	0.00 - 0.10	0.0042	52.4
0	0.10 - 0.20	N.A.	N.A.
0	0.20 - 0.50	N.A.	N.A.
0	0.50 - 1.00	N.A.	N.A.
111	Overall	0.0042	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	37	0.0055	0.0144

NITRILOTRIACETIC ACID (mg/L as NTA)
QUALITY CONTROL DATA FROM 21/01/99 TO 23/12/99
E3406



NITROGEN, AMMONIA PLUS AMMONIUM

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3364	Reporting Unit	mg/L as N
LIMS Product Code	DISNUT3364	Supervisor	P.Wilson
Sample Type/Matrix	Dried Sludge, Sediment, Soil, Vegetation, Drinking Water, Ground Water, Surface Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.

Approximate absorbance: 0.5 at the full scale level.

Nitrate plus nitrite, nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37°C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.002	Current T value: 0.01
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL, standard, and BL after every 10 samples

NOTES:

The HP data capture / processing system was replaced by Labtronics in August 1999.

NITROGEN, AMMONIA PLUS AMMONIUM (E3364)

QUALITY CONTROL DATA FROM 04/01/99 TO 23/12/99

Laboratory Unit: Colourimetry

Full Scale: to 2.00 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	107	1.60	1.606	0.006	0.0136
B:	107	0.800	0.803	0.003	0.0089
C:	107	0.160	0.1598	-0.0002	0.0058
A+B:		2.40	2.409	0.009	0.0162
A-B:		0.800	0.803	0.003	0.0163
B+C:		0.960	0.963	0.003	0.0125
B-C:		0.640	0.643	0.003	0.0085

s.d.(AB) S(between runs):0.0115

Sw(within run): 0.0115

S/Sw: 1.0

s.d.(BC) S(between runs):0.0076

Sw(within run): 0.0060

S/Sw: 1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

2.353 - 2.447 for A+B
0.765 - 0.835 for A-B
0.931 - 0.989 for B+C
0.618 - 0.662 for B-C

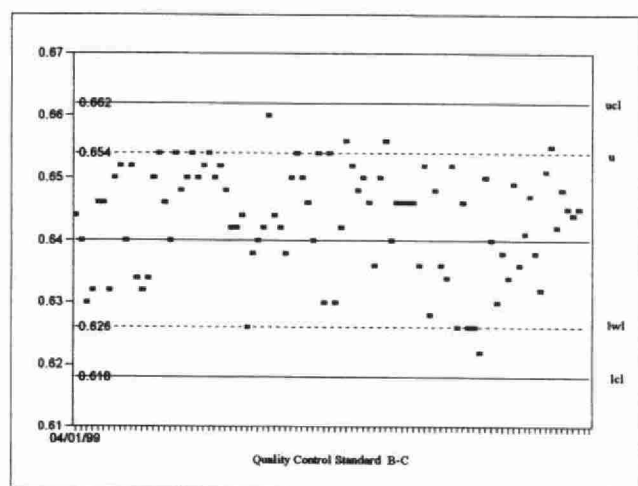
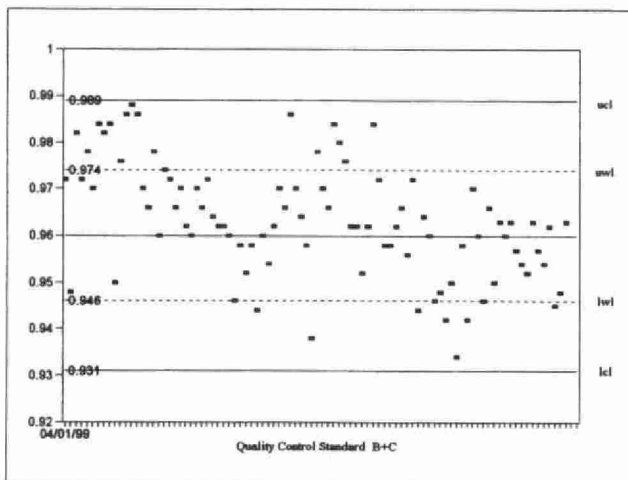
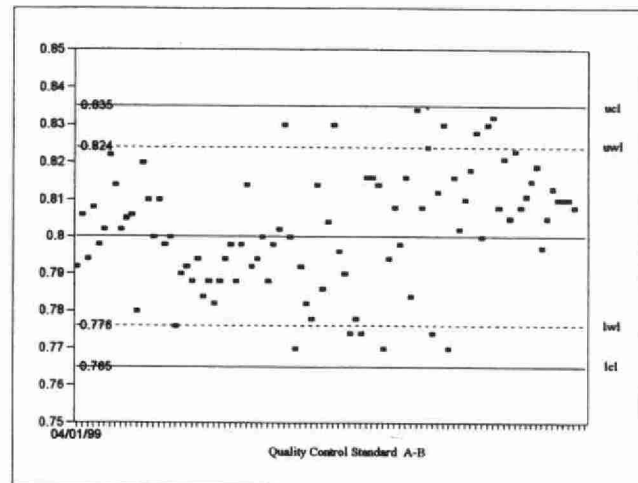
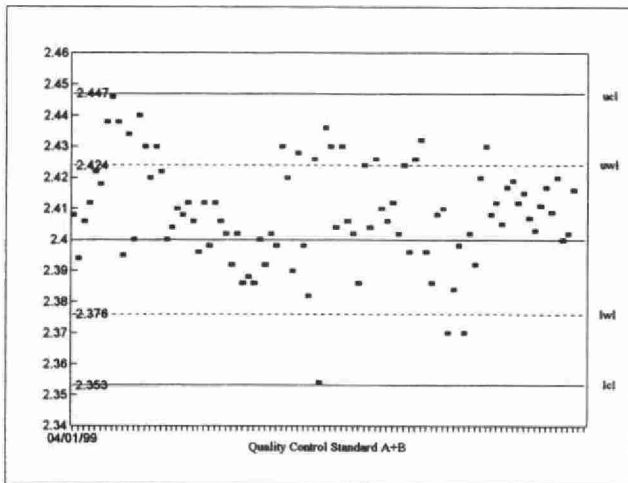
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
276	0 - 0.200	0.0055	18.8
14	0.201 - 0.400	0.0137	5.0
17	0.401 - 1.00	0.0095	1.3
3	1.01 - 2.00	0.0153	1.1
310	Overall	0.0079	8.8

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	107	0.0013	0.0044

NITROGEN, AMMONIA PLUS AMMONIUM (mg/l as N)
 QUALITY CONTROL DATA FROM 04/01/99 TO 23/12/99
 E3364



NITROGEN, AMMONIA PLUS AMMONIUM

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/77
Method Reference No.	E3366	Reporting Unit	mg/L as N
LIMS Product Code	DISNUT3366	Supervisor	P.Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Leachate, Drinking Water, Ground Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst.

Approximate absorbance: 0.7 at the full scale level.

Reactive orthophosphate, nitrogen-nitrite and nitrogen-nitrate plus nitrite are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus one 38°C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.05	Current T value: 0.25
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL, standard and BL every 10 samples

NOTES:

The HP capture / processing system was replaced by Labtronics in October 1999.

NITROGEN, AMMONIA PLUS AMMONIUM (E3366)

QUALITY CONTROL DATA FROM 05/01/99 TO 23/12/99

Laboratory Unit: Colourimetry

Full Scale: to 50.0 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	92	40.0	39.884	-0.116	0.3846
B:	92	20.0	19.959	-0.041	0.1981
C:	92	4.00	3.978	-0.022	0.1129
A+B:		60.0	59.843	-0.157	0.4950
A-B:		20.0	19.925	-0.075	0.3596
B+C:		24.0	23.937	-0.063	0.2539
B-C:		16.0	15.981	-0.019	0.1988

s.d.(AB) S(between runs):0.25

Sw(within run): 0.31

S/Sw: 1.2

s.d.(BC) S(between runs):0.14

Sw(within run): 0.16

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

58.76	-	61.24	for	A+B
19.07	-	20.93	for	A-B
23.34	-	24.66	for	B+C
15.50	-	16.50	for	B-C

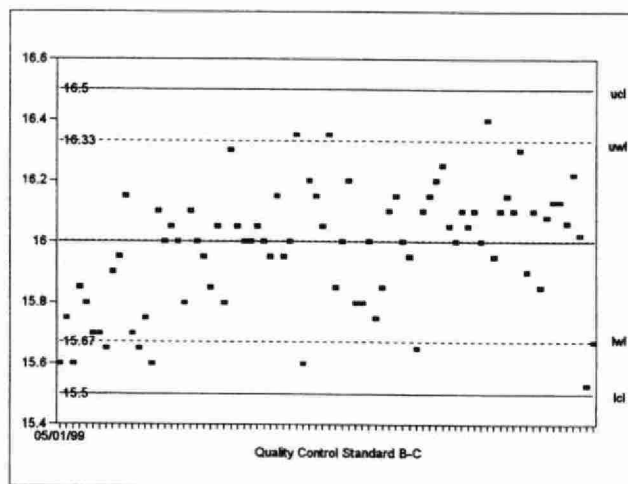
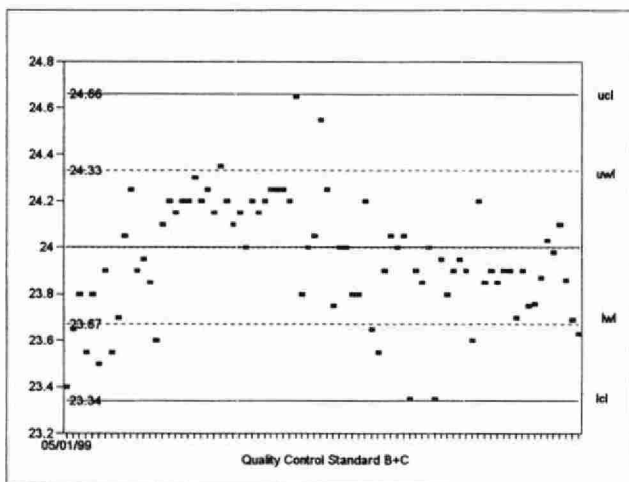
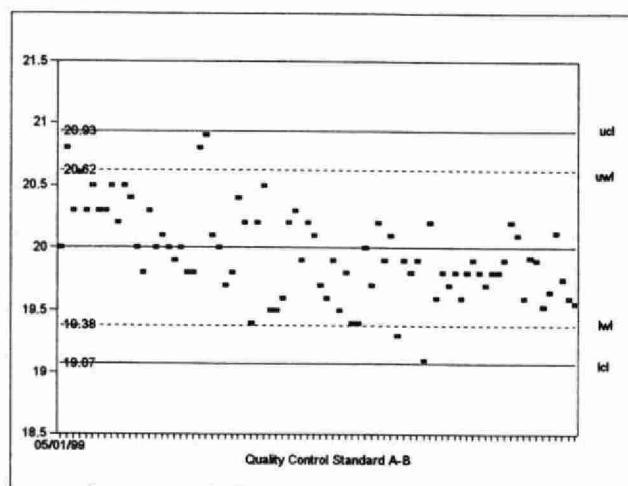
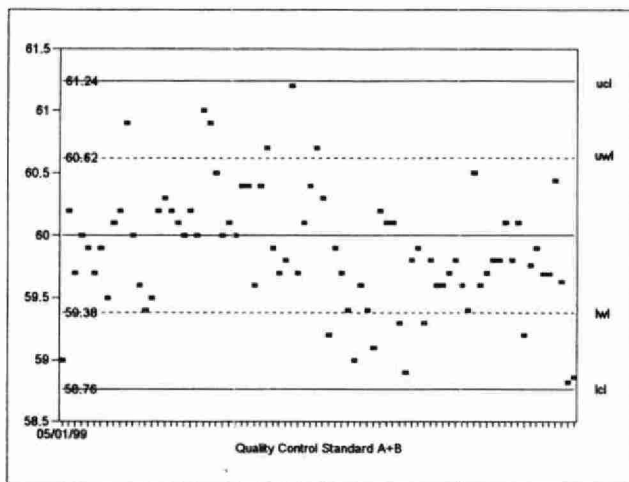
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
220	0.00 - 5.00	0.0634	16.8
17	5.10 - 10.0	0.1390	2.0
19	10.1 - 25.0	0.2588	1.6
12	25.1 - 50.0	0.1867	0.6
268	Overall	0.1044	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	92	0.0413	0.0965

NITROGEN, AMMONIA PLUS AMMONIUM (mg/L as N)
 QUALITY CONTROL DATA FROM 05/01/99 TO 23/12/99
 E3366



NITROGEN, AMMONIA PLUS AMMONIUM

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	01/06/76
Method Reference No.	E3374	Reporting Unit	µg/L as N
LIMS Product Code	AMMNO3374	Supervisor	F. Tomassini
Sample Type/Matrix:	Surface Waters, Precipitation, Leachate		

SAMPLING:

Quantity Required:	50 mL
Container:	Glass or plastic

ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.

Approximate absorbance : 0.40 at the full scale level.

Nitrate plus nitrite is determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37°C heating bath (7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm. Two analytical ranges are obtained from the output of the colourimeter.

REPORTING:

Maximum Significant Figures: 3	Current W value: 1	Current T value: 5
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CALIBRATION:

BL plus 8 standards

CONTROLS:

Calibration	LTBL plus 3 QC standards, e.g. QCA
Drift	BL every 10 samples and BL plus check standard every 20 samples

NITROGEN, AMMONIA PLUS AMMONIUM (E3374)

QUALITY CONTROL DATA FROM 21/01/99 TO 14/12/99

Full Scale: to 1000 µg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	18	600	598.83	-1.17	5.2608
B:	18	200	193.11	-6.89	2.5179
C:	18	60	60.83	0.83	1.5435
A+B:		800	791.94	-8.06	6.3660
A-B:		400	405.72	5.72	5.2447
B+C:		260	253.94	-6.06	3.1337
B-C:		140	132.28	-7.72	2.7612

s.d.(AB) S(between runs): 4.14

Sw(within run): 3.71

S/Sw: 1.1

s.d.(BC) S(between runs): 2.09

Sw(within run): 1.95

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

780	-	820	for	A+B
385	-	415	for	A-B
248	-	272	for	B+C
131	-	149	for	B-C

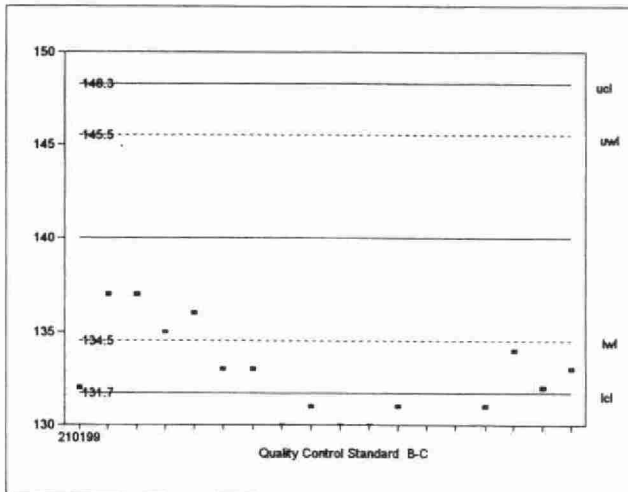
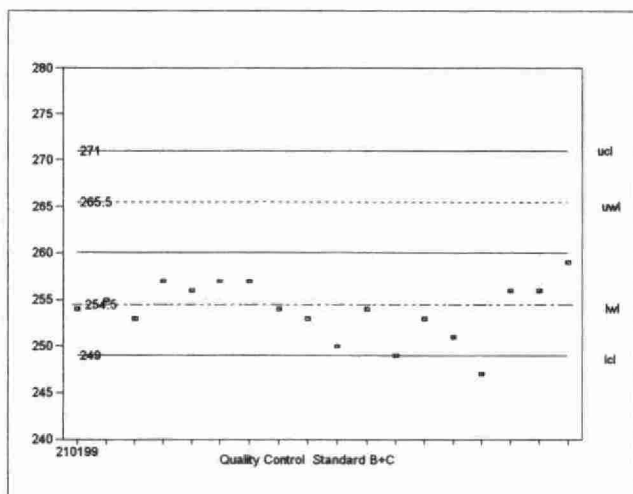
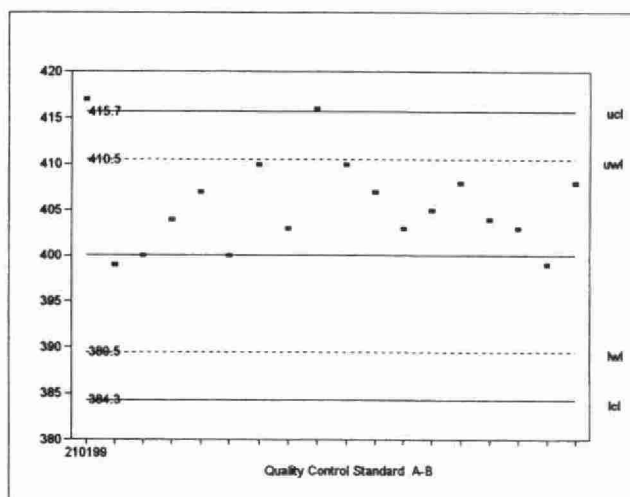
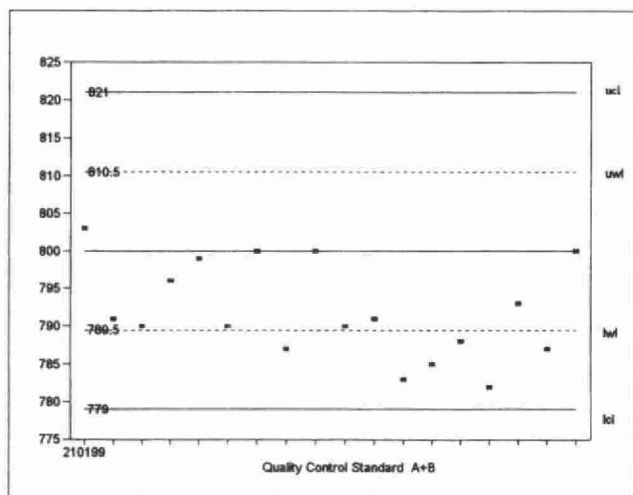
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
29	0.0 - 100	1.4622	6.5
14	101 - 500	5.3552	1.9
5	501 - 1000	4.7749	0.6
48	Overall	3.4686	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	18	1.3	0.9979

NITROGEN, AMMONIA PLUS AMMONIUM ($\mu\text{g/L}$ as N)
QUALITY CONTROL DATA FROM 21/01/99 TO 14/12/99
E3374



NITROGEN, NITRATE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/07/80
Method Reference No	E3148	Reporting Unit	µg/Filter as N
LIMS Product Code	LOV3148, ANLOV3148, NYL3148, TEF3148, ANION3148	Supervisor	P. Wilson
Sample Type/Matrix	Air; Sequential and LoVol filters		

SAMPLING:

Quantity Required	1 filter
Container	50 mL polypropylene tube

SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of Pure-DW (W40) or 25.0 mL of Pure-DW (Teflon) or 25.0 mL of 0.03N NaOH (Nylon) in polypropylene tubes with ultrasonic treatment followed by a 24 hour rest period.

ANALYTICAL PROCEDURE:

Nitrate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with $\text{Na}_2\text{CO}_3/\text{NaHCO}_3$ to match the eluent strength and maintain background conductivity. The concentration of nitrate in mg/L as N is determined by the comparison of the sample peak heights to a series of standards. Results are converted to µg/filter as N.

Chloride and sulphate are determined simultaneously.

INSTRUMENTATION:

Ultrasonic bath; modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing and partial data processing.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.01 mg/L	Current T value: 0.05 mg/L
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CALIBRATION:

BL plus 9 standards

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
Drift	1 standard every 10 samples

NOTES:

To convert unit from mg/L to µg/Filter, the concentration of N in mg/L is multiplied by 50 for W40 filters or 25 for Teflon and Nylon filters.

NITROGEN, NITRATE (E3148)

QUALITY CONTROL DATA FROM 26/01/99 TO 06/12/99

Full Scale: to 2.0 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	18	1.60	1.591	-0.009	0.0118
B:	18	0.40	0.398	-0.002	0.0226
A+B:		2.00	1.989	-0.011	0.0277
A-B:		1.20	1.193	-0.007	0.0230

s.d.(AB) S(between runs): 0.018 Sw(within run): 0.016 S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

1.93 - 2.07 for A+B
1.15 - 1.25 for A-B

DUPLICATES:

For W40 filters:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
13	0 - 0.40	0.0111	5.9
10	0.41 - 1.20	0.0225	4.5
3	1.21 - 2.00	0.0168	1.2
26	Overall	0.0170	

For Teflon filters:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
33	0 - 0.20	0.0104	35.5
1	0.21 - 0.40	N.A.	N.A.
3	0.41 - 1.20	0.0135	0.8
0	1.21 - 2.00	N.A.	N.A.
37	Overall	0.0106	

NITROGEN, NITRATE cont'd (E3148)

QUALITY CONTROL DATA FROM 26/01/99 TO 06/12/99

Full Scale: to 2.0 mg/L as N

DUPLICATES:

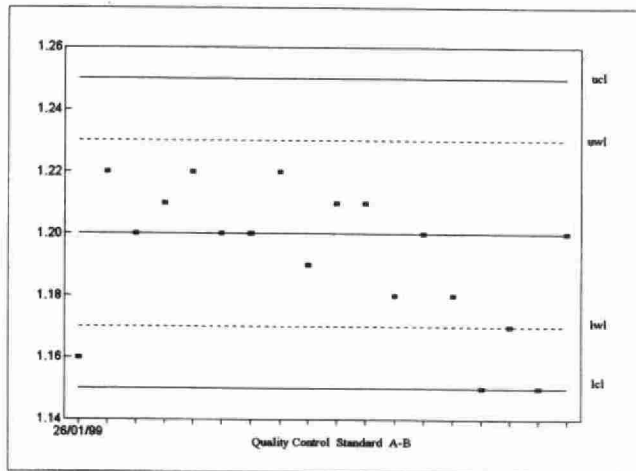
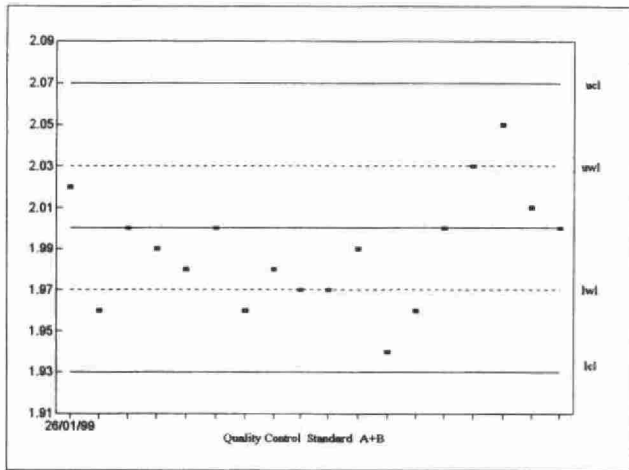
For Nylon filters:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
25	0 - 0.20	0.0063	10.5
11	0.21 - 0.40	0.0067	2.1
14	0.41 - 1.20	0.0132	2.4
0	1.21 - 2.00	N.A.	N.A.
50	Overall	0.0089	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	18	0.0011	0.0047

NITROGEN, NITRATE (mg/L as N)
 QUALITY CONTROL DATA FROM 26/01/99 TO 06/12/99
 E3148



NITROGEN, NITRATE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No	E3372	Reporting Unit	mg/L as N
LIMS Product Code	ANION3372	Supervisor	P. Wilson
Sample Type/Matrix	Precipitation		

SAMPLING:

Quantity Required	15 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Nitrate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with $\text{Na}_2\text{CO}_3/\text{NaHCO}_3$ to match the eluent strength and maintain background conductivity. The concentration of nitrate in mg/L as N is determined by the comparison of the sample peak heights to a series of standards.

Sulphate and chloride are determined simultaneously.

INSTRUMENTATION:

Modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing, and partial data processing.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.01	Current T value: 0.05
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
Drift	1 standard every 10 samples

NITROGEN, NITRATE (E3372)

QUALITY CONTROL DATA FROM 19/01/99 TO 23/12/99

Full Scale: to 1.0 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	20	0.80	0.808	0.008	0.0114
B:	20	0.20	0.191	-0.009	0.0162
A+B:		1.00	0.1004	0.0004	0.0186
A-B:		0.60	0.617	0.017	0.0210

s.d.(AB) S(between runs): 0.014 Sw(within run): 0.015 S/Sw: 0.95

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.96 - 1.04 for A+B
0.57 - 0.63 for A-B

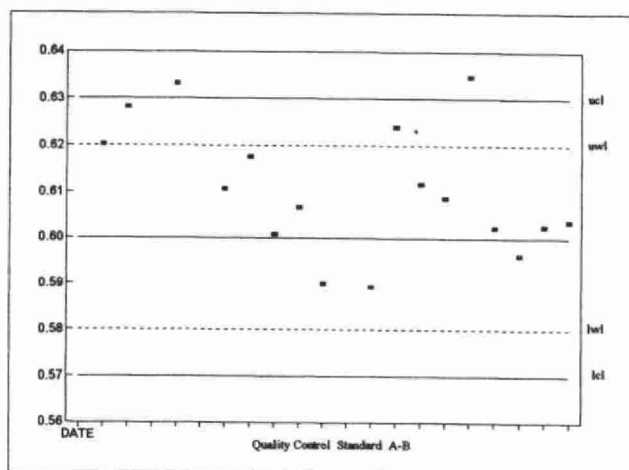
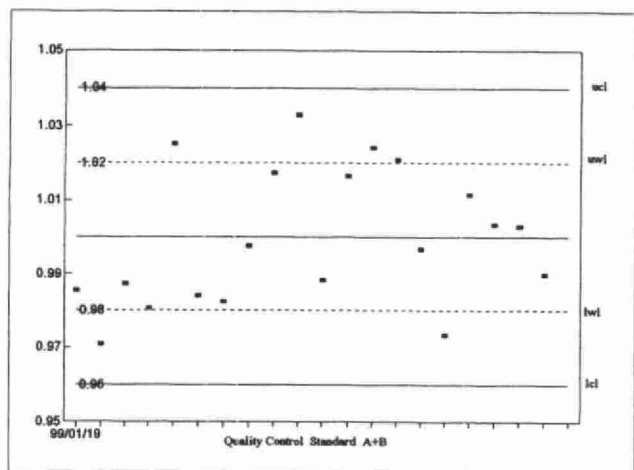
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
11	0 - 0.20	0.0116	25.1
19	0.21 - 0.50	0.0134	3.7
11	0.51 - 1.00	0.0209	2.8
41	Overall	0.0154	4.0

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	18	0	0

NITROGEN, NITRATE (mg/L as N)
QUALITY CONTROL DATA FROM 19/01/99 TO 23/12/99
E3372



NITROGEN, NITRATE PLUS NITRITE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3364	Reporting Unit	mg/L as N
LIMS Product Code	DISNUT3364	Supervisor	P.Wilson
Sample Type/Matrix	Dried Sludge, Sediment, Soil, Vegetation, Drinking Water, Ground Water, Surface Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 37°C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-naphthyl) ethylenediamine dihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step. Approximate absorbance: 0.6 at the full scale level.

Ammonia plus ammonium, nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37°C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 1.5 cm. light path at 520 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.005	Current T value: 0.025
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL, standard and BL every 10 samples
Interference	Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective interference suppression.
Recovery	Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step.

NOTES:

The HP data capture / processing system was replaced by Labtronics in August 1999.

NITROGEN, NITRATE PLUS NITRITE (E3364)

QUALITY CONTROL DATA FROM 04/01/99 TO 23/12/99

Laboratory Unit: Colourimetry

Full Scale: to 5.00 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	108	4.00	4.014	0.014	0.0360
B:	108	2.00	2.015	0.015	0.0221
C:	108	0.40	0.403	0.003	0.0077
A+B:		6.00	6.028	0.028	0.0422
A-B:		2.00	1.999	-0.001	0.0423
B+C:		2.40	2.418	0.018	0.0247
B-C:		1.60	1.611	0.011	0.0220

s.d.(AB) S(between runs):0.0300

Sw(within run): 0.0299

S/Sw: 1.0

s.d.(BC) S(between runs):0.0166

Sw(within run): 0.0157

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

5.868	-	6.132	for	A+B
1.901	-	2.099	for	A-B
2.328	-	2.472	for	B+C
1.546	-	1.654	for	B-C

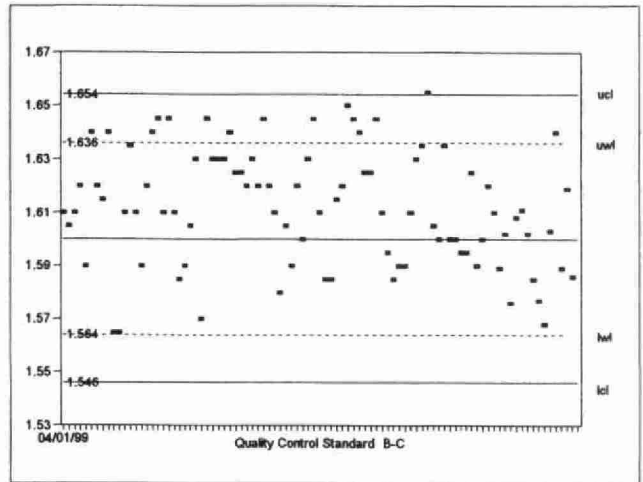
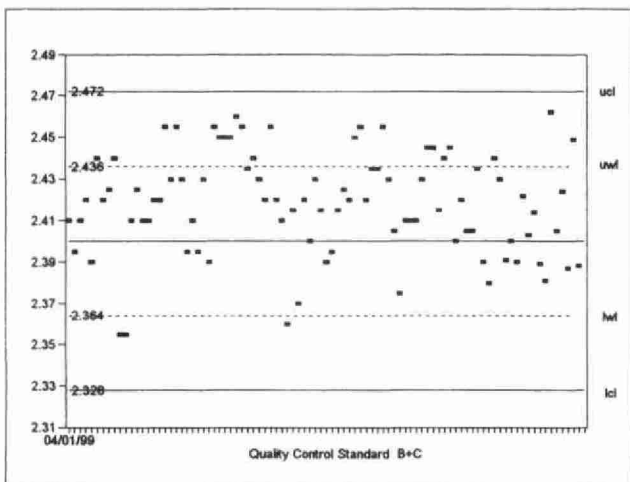
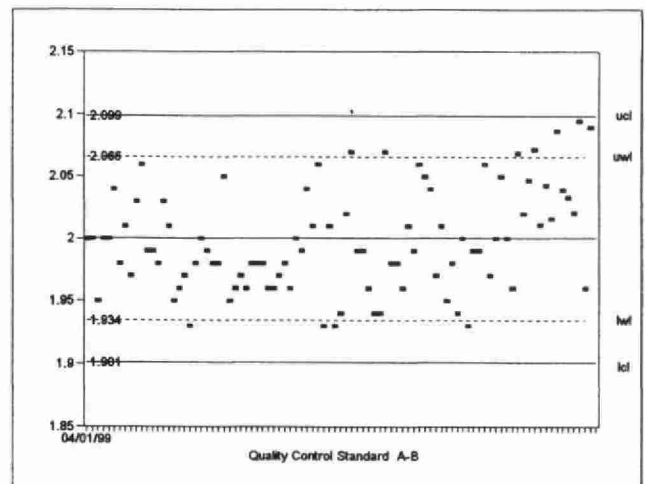
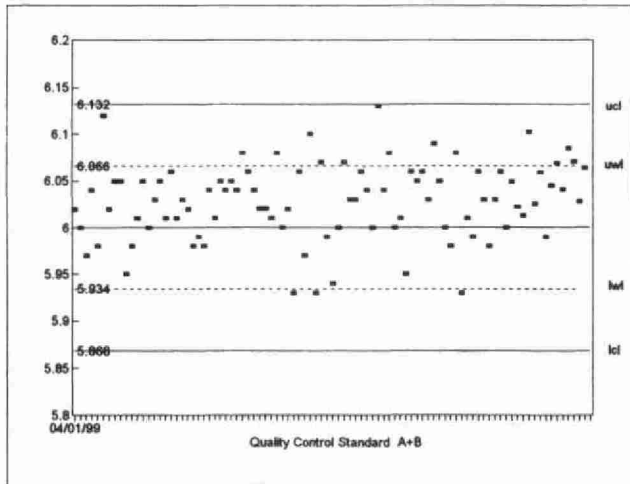
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
138	0 - 0.500	0.0054	9.8
67	0.501 - 1.00	0.0064	2.1
47	1.01 - 2.50	0.0183	3.0
32	2.51 - 5.00	0.0297	2.2
284	Overall	0.0134	3.8

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	108	0.003	0.0069

NITROGEN, NITRATE PLUS NITRITE (mg/L as N)
QUALITY CONTROL DATA FROM 04/01/99 TO 23/12/99
E3364



NITROGEN, NITRATE PLUS NITRITE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3366	Reporting Unit	mg/L as N
LIMS Product Code	DISNUT3366	Supervisor	P.Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Leachate, Drinking Water, Ground Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 38°C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-naphthyl) ethylenediamine dihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step. Approximate absorbance: 0.7 at the full scale level.

Ammonia plus ammonium, nitrite, and reactive phosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 38°C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 520 nm. Two analytical ranges are obtained from the output of the colourimeter. Data capture, reduction, and processing via a multi - stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.05	Current T value: 0.25
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL ,standard and BL every 10 samples
Interference	Nitrate standard spiked with calcium (150 mg/L) and magnesium (50mg/L) confirms effective interference suppression.
Recovery	Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step.

NOTES:

The HP capture / processing system was replaced by Labtronics in October 1999.

NITROGEN, NITRATE PLUS NITRITE (E3366)

QUALITY CONTROL DATA FROM 05/01/99 TO 23/12/99

Laboratory Unit: Colourimetry

Full Scale: to 50.0 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	94	40.0	39.934	-0.066	0.3820
B:	94	20.0	20.029	0.029	0.1994
C:	94	4.00	3.981	-0.019	0.0690
A+B:		60.0	59.962	-0.038	0.4735
A-B:		20.0	19.905	-0.095	0.3837
B+C:		24.0	24.009	0.009	0.2149
B-C:		16.0	16.044	0.044	0.2102

s.d.(AB) S(between runs):0.27

Sw(within run): 0.30

S/Sw: 1.1

s.d.(BC) S(between runs):0.15

Sw(within run): 0.15

S/Sw: 1.0

The calibration is accepted if the calibration control values obtained lie within the ranges:

58.76	-	61.24	for	A+B
19.07	-	20.93	for	A-B
23.34	-	24.66	for	B+C
15.50	-	16.50	for	B-C

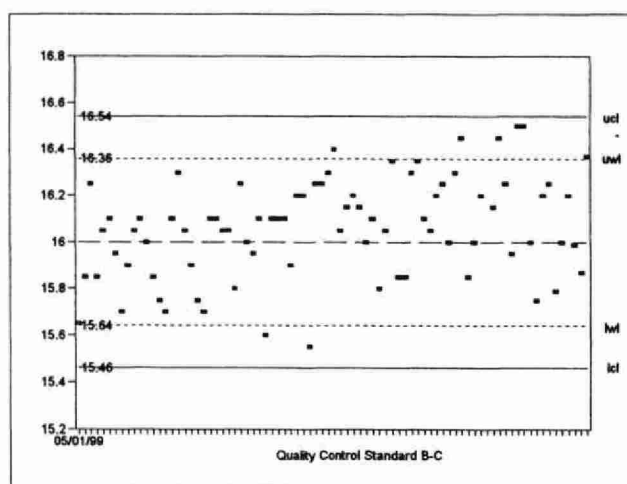
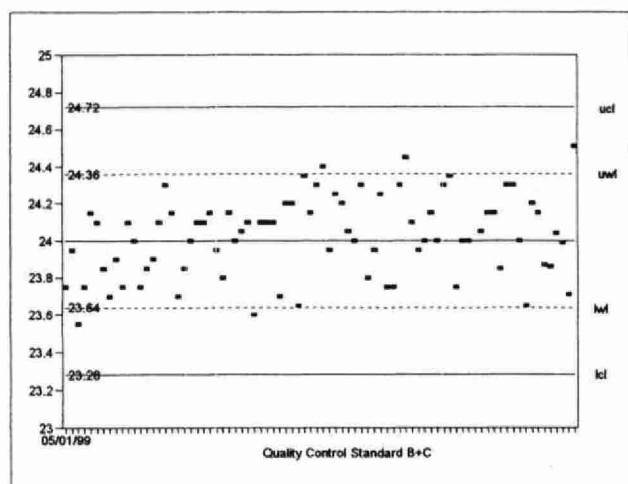
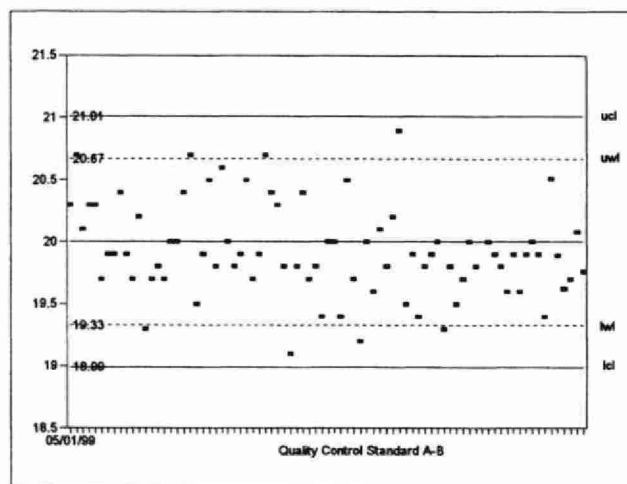
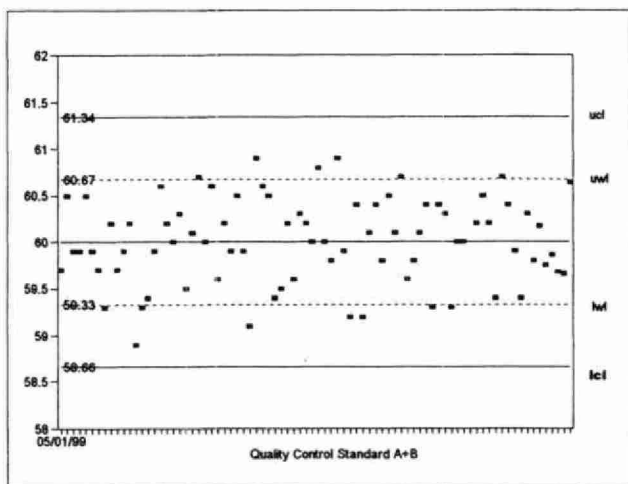
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
183	0.00 - 5.00	0.0622	6.4
32	5.10 - 10.0	0.1759	2.4
51	10.1 - 25.0	0.3109	2.0
11	25.1 - 50.0	0.3900	1.1
277	Overall	0.1731	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	94	0.0138	0.0748

NITROGEN, NITRATE PLUS NITRITE (mg/L as N)
QUALITY CONTROL DATA FROM 05/01/99 TO 23/12/99
E3366



NITROGEN, NITRATE PLUS NITRITE

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	13/06/78
Method Reference No.	E3374	Reporting Unit	µg/L as N
LIMS Product Code	AMMNO3374	Supervisor	F. Tomassini
Sample Type/Matrix:	Surface Waters, Precipitation, Leachate		

SAMPLING:

Quantity Required:	50 mL
Container:	Glass or plastic

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a sample. Nitrate is reduced to nitrite in alkaline media at 37°C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-naphthyl) ethylenediamine dihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step.

Approximate absorbance : 0.4 at the full scale level.

Ammonia plus ammonium is determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37°C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 5.0 cm. light path at 520 nm.

REPORTING:

Maximum Significant Figures: 3	Current W value: 2	Current T value: 10
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CALIBRATION:

BL plus 8 standards

CONTROLS:

Calibration	LTBL plus 3 QC standards, e.g. QCA
Drift	BL every 10 samples and BL plus check standard every 20 samples

NITROGEN, NITRATE PLUS NITRITE (E3374)

QUALITY CONTROL DATA FROM 21/01/99 TO 14/12/989

Full Scale: to 1000 µg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	18	600	601.1	1.1	3.9329
B:	18	200	200.6	0.6	3.2217
C:	18	60	60.7	0.7	2.4495
A+B:		800	801.6	1.6	6.1753
A-B:		400	400.5	0.5	3.6822
B+C:		260	261.2	1.2	5.0475
B-C:		140	139.9	-0.1	2.6983

s.d.(AB) S(between runs): 3.60

Sw(within run): 2.60

S/Sw: 1.4

s.d.(BC) S(between runs): 2.86

Sw(within run): 1.91

S/Sw: 1.5

The calibration is accepted if the calibration control values obtained lie within the ranges:

780	-	820	for	A+B
385	-	415	for	A-B
248	-	272	for	B+C
131	-	149	for	B-C

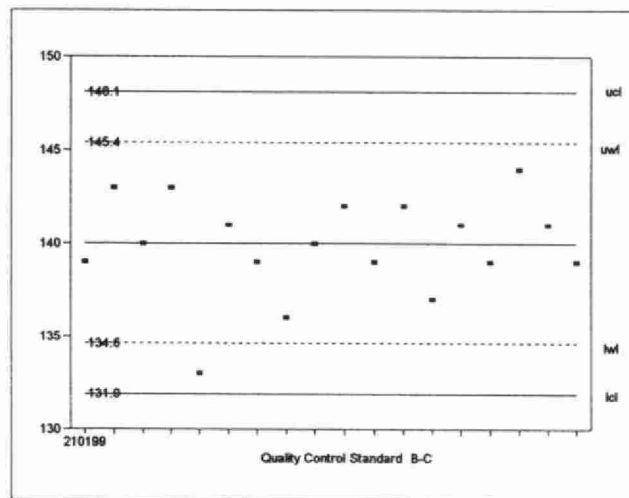
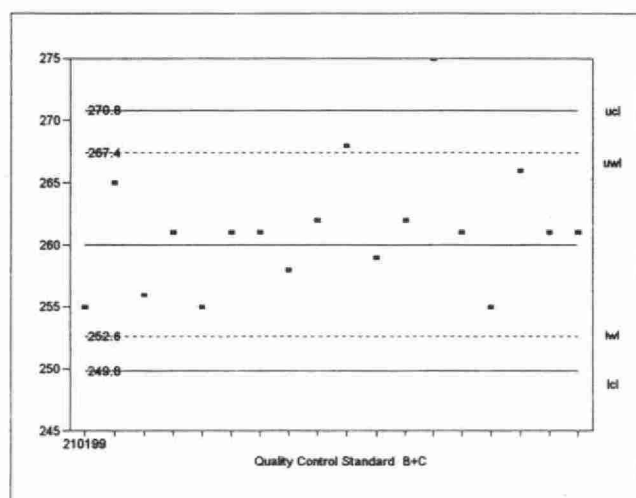
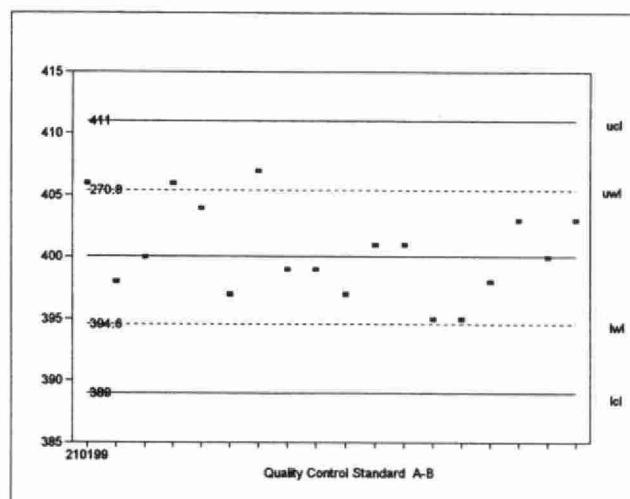
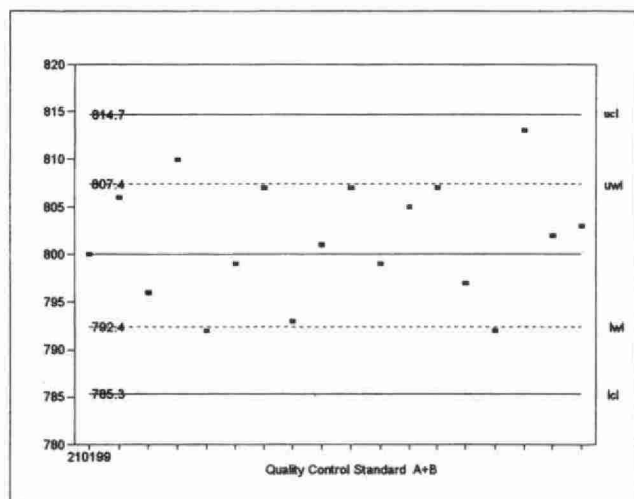
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
24	0.0 - 100	2.2958	6.9
16	101 - 500	3.5663	1.6
5	501 - 1000	8.2098	1.1
45	Overall	3.8500	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	18	0.67	0.9701

NITROGEN, NITRATE PLUS NITRITE ($\mu\text{g/L}$ as N)
QUALITY CONTROL DATA FROM 21/01/99 TO 14/12/99
E3374



NITROGEN, NITRITE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3364	Reporting Unit	mg/L as N
LIMS Product Code	DISNUT3364	Supervisor	P.Wilson
Sample Type/Matrix	Dried Sludge, Sediment, Soil, Vegetation, Drinking Water, Ground Water, Surface Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide, and N(1-naphthyl) ethylenediamine dihydrochloride.

Approximate absorbance: 0.6 at the full scale level.

Ammonia plus ammonium, nitrate plus nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.001	Current T value: 0.005
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL, standard and BL after every 10 samples
Interference	Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective interference suppression.
Recovery	Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step.

NOTES:

The HP data capture / processing system was replaced by Labtronics in August 1999.

NITROGEN, NITRITE (E3364)

QUALITY CONTROL DATA FROM 04/01/99 TO 23/12/99

Laboratory Unit: Colourimetry

Full Scale: to 0.200 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	107	0.160	0.15995	-0.00005	0.0017
B:	107	0.080	0.0799	-0.0001	0.0012
C:	107	0.016	0.01605	0.00005	0.0006
A+B:		0.240	0.2398	-0.0002	0.0024
A-B:		0.080	0.0801	0.0001	0.0017
B+C:		0.096	0.0959	-0.0001	0.0015
B-C:		0.064	0.0638	-0.0002	0.0012

s.d.(AB) S(between runs):0.0015

Sw(within run): 0.0012

S/Sw: 1.2

s.d.(BC) S(between runs):0.00097

Sw(within run): 0.00085

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.234	-	0.246	for	A+B
0.076	-	0.084	for	A-B
0.092	-	0.100	for	B+C
0.061	-	0.067	for	B-C

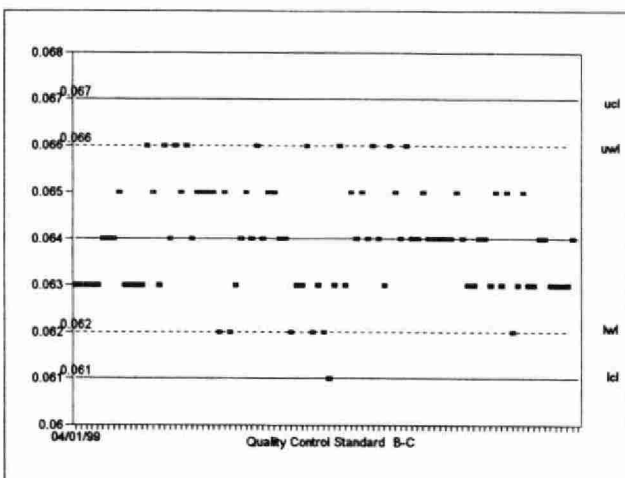
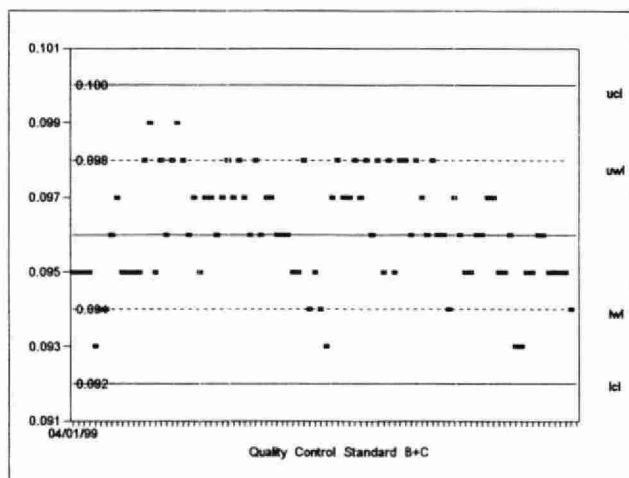
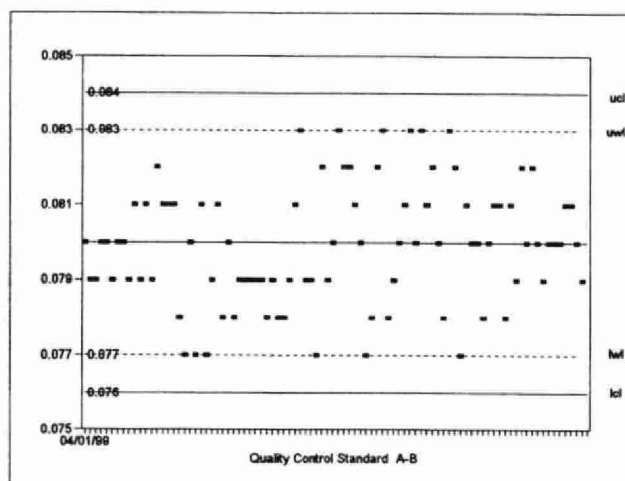
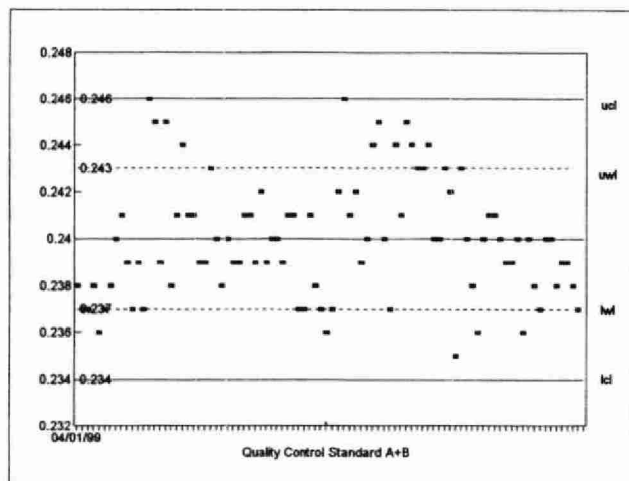
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
267	0 - 0.020	0.0013	35.7
19	0.021 - 0.040	0.0011	3.8
18	0.041 - 0.100	0.0030	4.3
2	0.101 - 0.200	0.0020	1.5
306	Overall	0.0015	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	107	0.0004	0.0009

NITROGEN, NITRITE (mg/L as N)
QUALITY CONTROL DATA FROM 04/01/99 TO 23/12/99
E3364



NITROGEN, NITRITE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No	E3366	Reporting Unit	mg/L as N
LIMS Product Code	DISNUT3366	Supervisor	P.Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Leachate, Drinking Water, Ground Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide, and N(1-naphthyl) ethylenediamine dihydrochloride.

Approximate absorbance: 0.3 at the full scale level.

Ammonia plus ammonium, nitrate plus nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.005	Current T value: 0.025
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL ,standard and BL every 10 samples
Interference	Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective interference suppression.
Recovery	Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step.

NOTES:

The HP capture / processing system was replaced by Labtronics in October 1999.

NITROGEN, NITRITE (E3366)

QUALITY CONTROL DATA FROM 05/01/99 TO 23/12/99

Laboratory Unit: Colourimetry

Full Scale: to 2.00 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	93	1.60	1.61	0.01	0.0150
B:	93	0.80	0.8005	0.0005	0.0092
C:	93	0.160	0.159	-0.001	0.0051
A+B:		2.40	2.413	0.013	0.0188
A-B:		0.80	0.812	0.012	0.0163
B+C:		0.960	0.9603	0.0003	0.0112
B-C:		0.640	0.641	0.001	0.0097

s.d.(AB) S(between runs):0.0124

s.d.(BC) S(between runs):0.0074

Sw(within run): 0.0116

Sw(within run): 0.0069

S/Sw: 1.1

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

2.352 - 2.448 for A+B
 0.764 - 0.836 for A-B
 0.936 - 0.984 for B+C
 0.622 - 0.658 for B-C

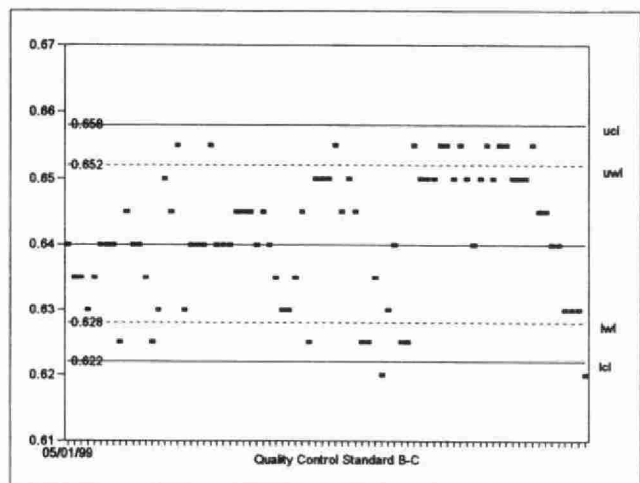
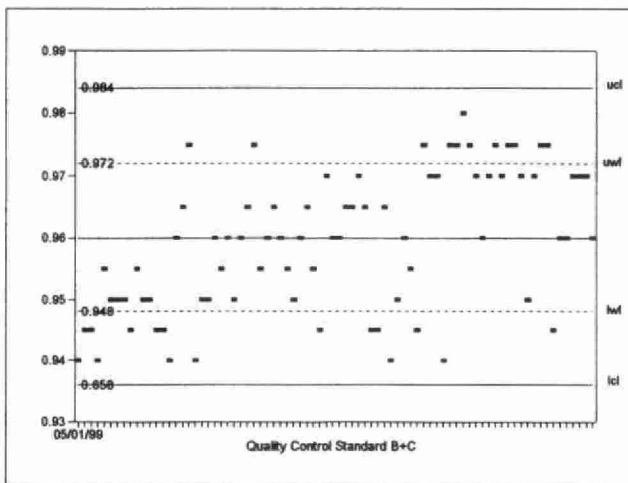
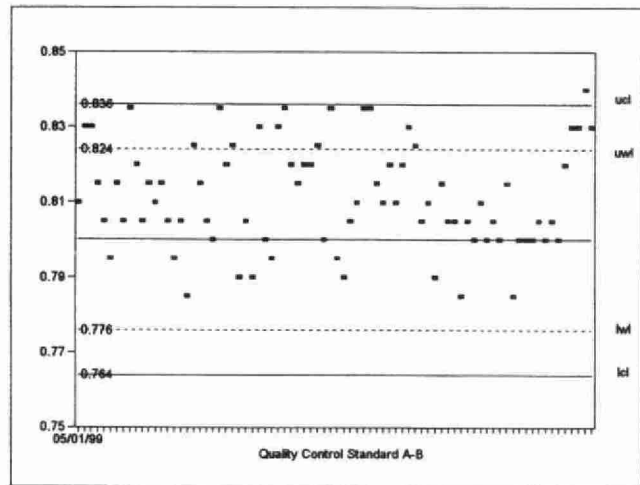
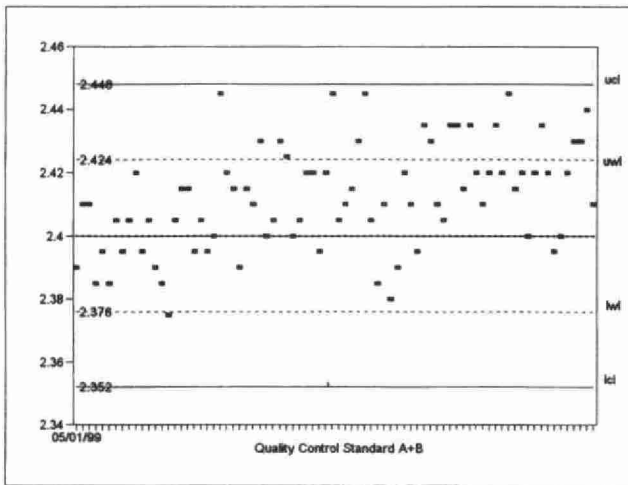
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
215	0.000 - 0.200	0.0066	23.0
25	0.201 - 0.400	0.0135	5.0
20	0.401 - 1.00	0.0170	2.5
13	1.01 - 2.00	0.0629	1.1
273	Overall	0.0162	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	93	-0.0018	0.0044

NITROGEN. NITRITE (mg/L as N)
QUALITY CONTROL DATA FROM 05/01/99 TO 23/12/99
E3366



NITROGEN, TOTAL KJELDAHL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Mar '89
Method Reference No.	E3116	Reporting Unit	mg/g as N
LIMS Product Code	TNP3116	Supervisor	P. Wilson
Sample Type/Matrix	Soil, Sediment, Dried Sludge		

SAMPLING:

Quantity Required	0.08 to 0.4 g
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Nitrogen compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is filtered and the filtrate is analyzed using an automated colourimetric system.

INSTRUMENTATION:

Hot plate .

Basic automated modular continuous flow system : 37.5°C bath. Colourimetric measurement is through a 5 cm. light path at 630 nm.

Data capture, and processing via a microcomputer system.

REPORTING:

Maximum Significant Figures: 2 decimal places	Current W value: 0.1	Current T value: 0.5
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CALIBRATION:

3 High and 2 Low Calibration Standards

CONTROLS:

Drift	4 BL's per run; high and low calibration standard at the end of the run
Recovery	2 digested standards (R1,R2) to monitor digestion procedure, a certified reference material RSM2781 to monitor digestion procedure for sludge samples and an in house composite (RS92) to monitor digestion procedure for sediment and soils.

NOTES:

System is calibrated with undigested standards. Recovery Standards R1 and R2 are standards prepared in Pure Water and are less complex than soil and sludge matrices. R1 and R2 showed levels within limits for 1999 with 4 exceptions (May 14, Aug 18, Sept 29, and Nov 22) ranging from $\pm 12\%$ for R1 and one exception for R2 on Aug 6 at 11%. RS92 showed the lower limit was exceeded by -20% and the RSM2781 showed both some high (up to 11%) but generally low recoveries of -19 %. The digestion procedure is still under investigation.

NITROGEN, TOTAL KJELDAHL (E3116)

QUALITY CONTROL DATA FROM 07/04/99 TO 03/12/99

Full Scale: 10 mg/g as N

QUALITY CONTROL:

	n	Expected Concentration	Mean Concentration	Standard Deviation (1)
RS92 - In House Soil Composite	34	1.69	1.39	0.2327
RSM-2781 -Reference Standard Material	34	47.8	43.39	5.8045

The run is accepted if the control values obtained lie within the ranges:

1.39 - 1.99 for RS92
46.7 - 48.9 for RSM-2781

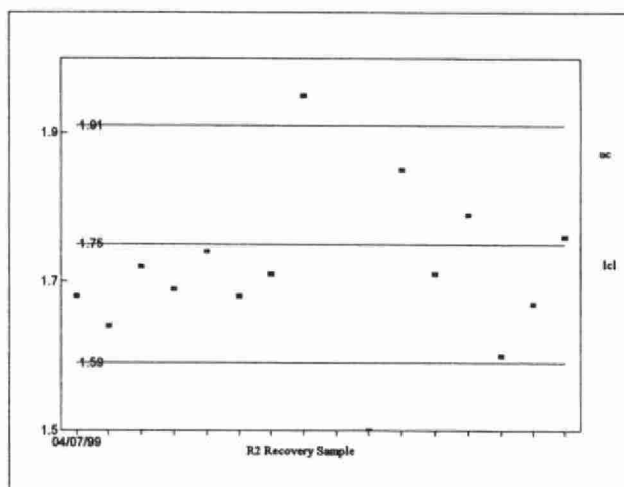
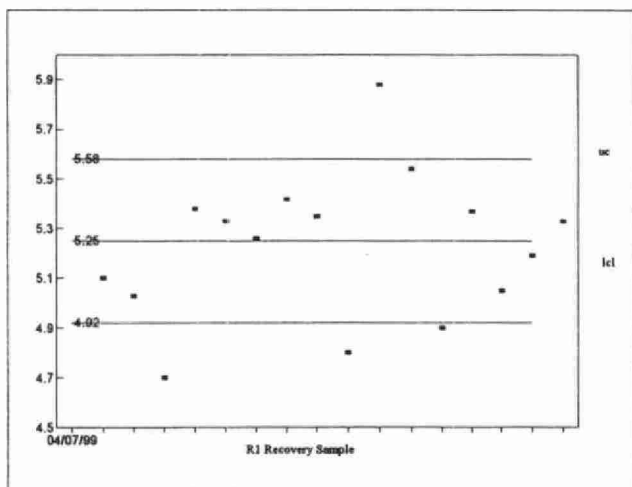
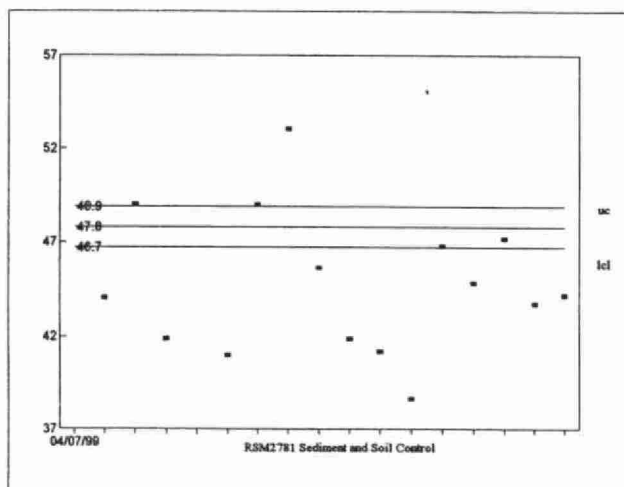
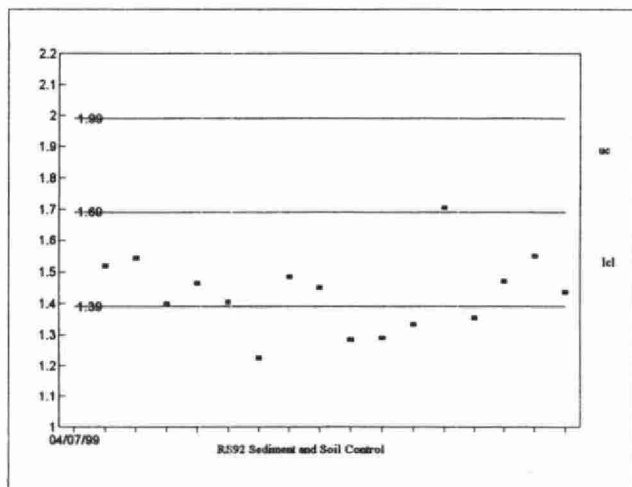
Recovery Standards

	n	Expected Concentration	Mean Concentration	Standard Deviation (1)
R1	16	5.25	5.227	0.2941
R2	16	1.75	1.669	0.1150

DUPLICATES: (Sediment/Soils)

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
14	0.00 - 2.00	0.0852	7.8
6	2.01 - 4.00	0.1918	7.5
3	4.01 - 10.0	0.2770	5.8
4	10.1 - 20.0	0.4783	3.3
43	Overall	0.1919	6.9

NITROGEN, TOTAL KJELDAHL (mg/g as N)
QUALITY CONTROL DATA FROM 07/04/99 TO 03/12/99
E3116



NITROGEN, TOTAL KJELDAHL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Mar '89
Method Reference No.	E3118	Reporting Unit	mg/g as N
LIMS Product Code	TNP3118	Supervisor	P. Wilson
Sample Type/Matrix	Vegetation, Moss Bag		

SAMPLING:

Quantity Required	0.02 to 0.04 g
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Nitrogen compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is analyzed using an automated colourimetric system.

INSTRUMENTATION:

Hot plate.

Basic automated modular continuous flow system : 37.5°C bath. Colourimetric measurement is through a 5 cm. light path at 630 nm.

Data capture, and processing via a microcomputer system.

REPORTING:

Maximum Significant Figures: 2 decimal places	Current W value: 0.20	Current T value: 1.00
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CALIBRATION:

3 High and 2 Low Calibration Standards

CONTROLS:

Calibration	In house composite A-VEG, plus QC VEG (Pine Needles)
Drift	4 BL's per run; high and low calibration standard at the end of the run
Recovery	1 digested BL plus 4 digested standards

NOTES:

System is calibrated with undigested standards.

There is not enough data to report QC for 1999.

NITROGEN, TOTAL KJELDAHL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/79
Method Reference No.	E3367	Reporting Unit	mg/L as N
LIMS Product Code	TOTNUT3367	Supervisor	P.Wilson
Sample Type/Matrix	Precipitation, Drinking Water, Ground Water , Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digestors kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst.

Approximate absorbance: 0.3 at the full scale level.

Total phosphorus is determined simultaneously.

INSTRUMENTATION:

Three block digesters

Basic automated modular continuous flow system plus 1 module: 38°C bath (7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.02	Current T value: 0.1
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CALIBRATION:

BL plus 7 undigested standards

CONTROLS:

Calibration	LTBL plus 3 undigested standards, e.g. QCA
Drift	BL, undigested standard , BL every 10 samples
Recovery	3 digested BL plus 3 digested standards in duplicate, e.g. R1

NOTE:

The HP capture / processing system was replaced by Labtronics in May 1999

NITROGEN, TOTAL KJELDAHL (E3367)

QUALITY CONTROL DATA FROM 11/01/99 TO 16/12/99

Full Scale: to 2.00 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	111	1.60	1.603	0.003	0.0160
B:	111	0.800	0.806	0.006	0.0103
C:	111	0.160	0.161	0.001	0.0102
A+B:		2.40	2.409	0.009	0.0210
A-B:		0.800	0.797	-0.003	0.0167
B+C:		0.960	0.967	0.007	0.0148
B-C:		0.640	0.645	0.005	0.0143

s.d.(AB) S(between runs): 0.013

Sw(within run): 0.012

S/Sw: 1.1

s.d.(BC) S(between runs): 0.010

Sw(within run): 0.010

S/Sw: 1.0

The calibration is accepted if the calibration control values obtained lie within the ranges:

2.32 - 2.48 for A+B
 0.740 - 0.860 for A-B
 0.913 - 1.007 for B+C
 0.605 - 0.675 for B-C

RECOVERIES:

Number of Data	Expected Concentration	Mean Concentration	Standard Deviation (1)
111	1.40	1.392	0.0375
111	0.840	0.827	0.0322
111	0.280	0.276	0.0209

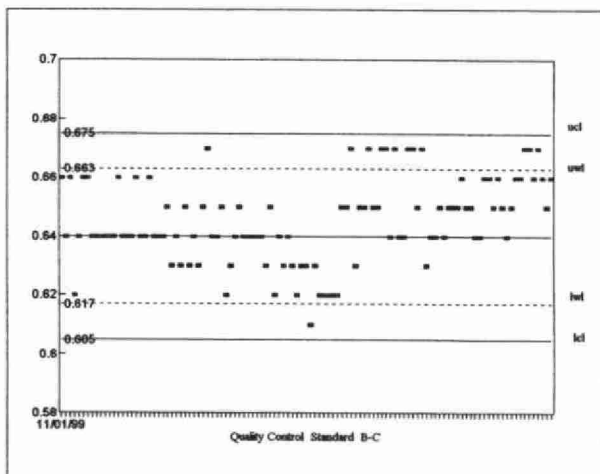
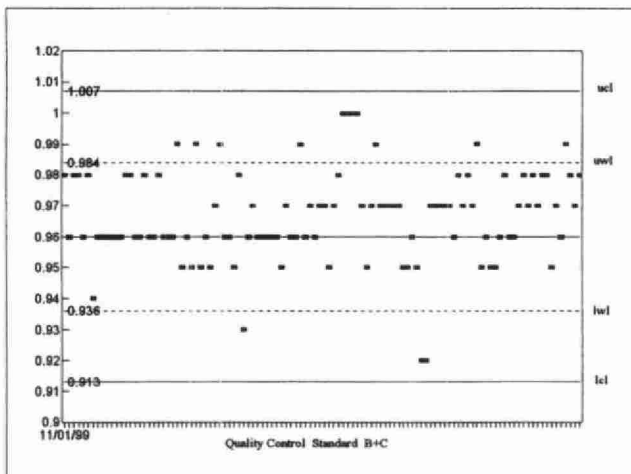
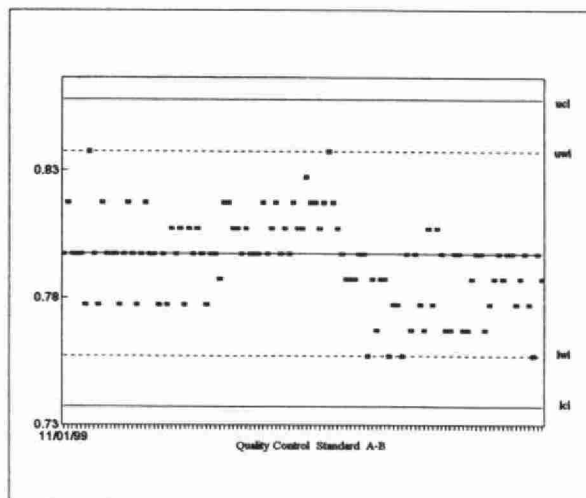
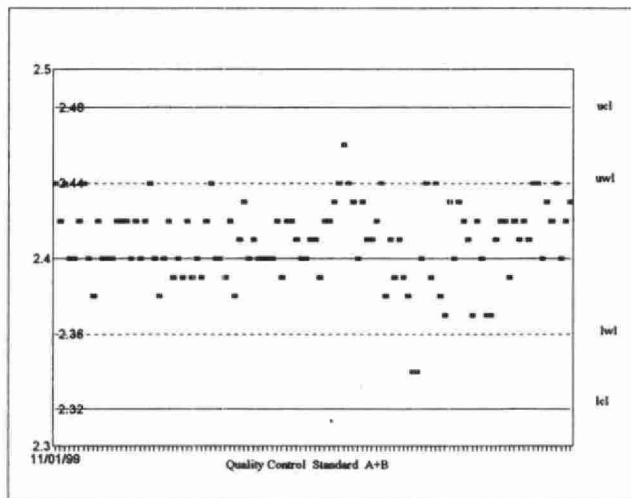
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
81	0 - 0.200	0.0358	23.6
113	0.201 - 0.400	0.0880	28.6
93	0.401 - 1.00	0.0850	14.5
6	1.01 - 2.00	0.4220	28.2
293	Overall	0.0964	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	111	0.0051	0.0235
Digested Blank	111	-0.0003	0.0164

NITROGEN, TOTAL KJELDAHL (mg/L as N)
 QUALITY CONTROL DATA FROM 11/01/99 TO 16/12/99
 E3367



NITROGEN, TOTAL KJELDAHL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/79
Method Reference No	E3368	Reporting Unit	mg/L as N
LIMS Product Code	TOTNUT3368	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Raw Sewage, Industrial Waste, Drinking Water, Effluent, Ground Water, Process Water, Leachate		

SAMPLING:

Quantity Required	50 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digestors kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst.

Approximate absorbance: 1.1 at the full scale level.

Total phosphorus is determined simultaneously.

INSTRUMENTATION:

Three block digesters

Basic automated modular continuous flow system plus 1 module: 38°C bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.05	Current T value: 0.25
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CALIBRATION:

BL plus 7 undigested standards

CONTROLS:

Calibration	LTBL plus 3 undigested standards, e.g. QCA
Drift	BL every 10 samples; undigested standard every 20 samples
Recovery	3 digested BL plus 3 digested standards in duplicate, e.g. R1

NOTES:

System is calibrated with undigested standards.

The HP capture / processing system was replaced by Labtronics in April 1999

NITROGEN, TOTAL KJELDAHL (E3368)

QUALITY CONTROL DATA FROM 10/01/99 TO 29/12/99

Full Scale: to 50.0 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	47	40.0	40.006	0.006	0.1466
B:	47	20.0	20.023	0.023	0.1042
C:	47	4.0	4.027	0.027	0.0684
A+B:		60.0	60.030	0.030	0.2007
A-B:		20.0	19.983	-0.017	0.1564
B+C:		24.0	24.050	0.050	0.1348
B-C:		16.0	15.996	-0.004	0.1136

s.d.(AB) S(between runs): 0.127

Sw(within run): 0.111

S/Sw: 1.15

s.d.(BC) S(between runs): 0.088

Sw(within run): 0.080

S/Sw: 1.10

The calibration is accepted if the calibration control values obtained lie within the ranges:

59.27	-	60.73	for	A+B
19.45	-	20.55	for	A-B
23.58	-	24.42	for	B+C
15.68	-	16.32	for	B-C

RECOVERIES:

Number of Data	Expected Concentration	Mean Concentration	Standard Deviation (1)
46	35	34.85	0.7275
45	21	20.68	0.9051
46	7	6.88	0.2232

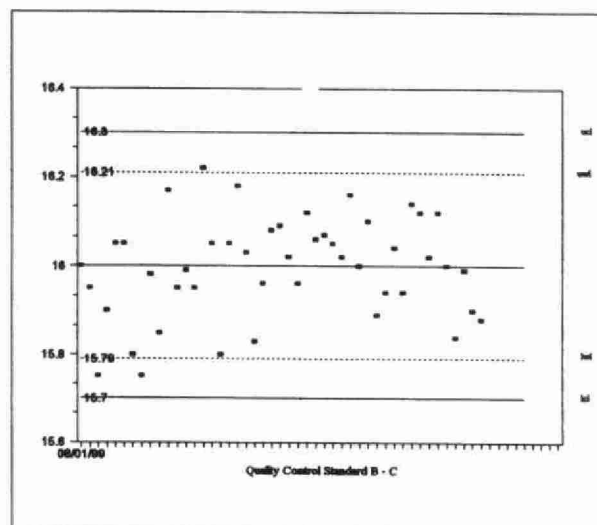
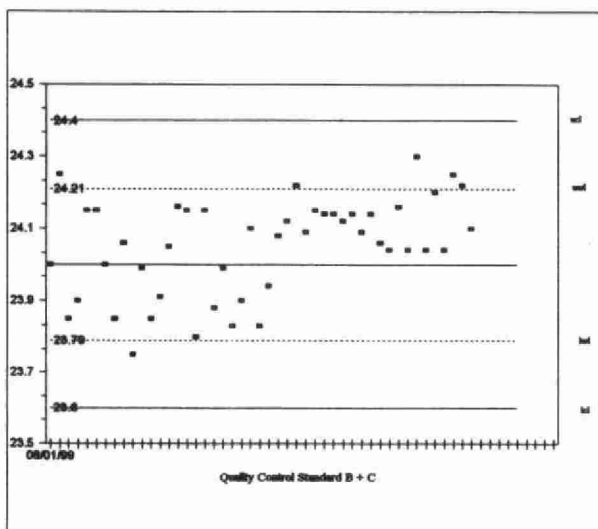
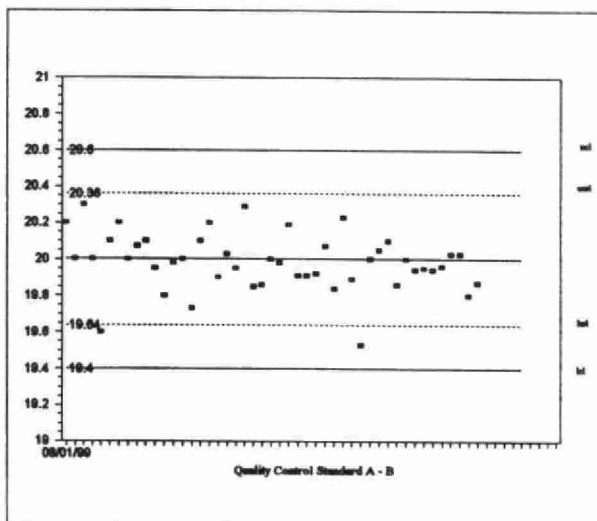
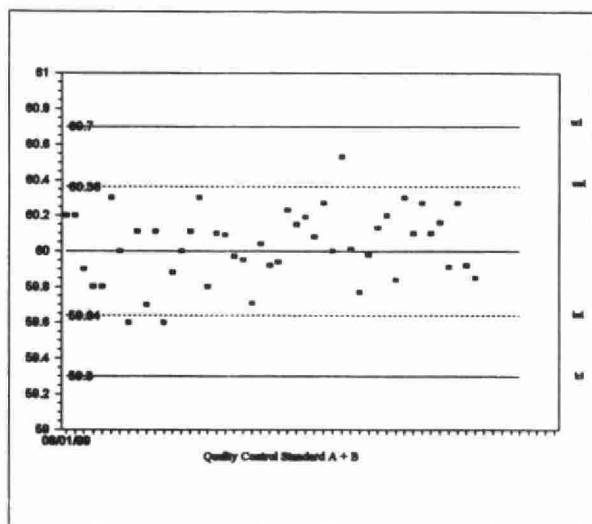
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
97	0.00 - 5.00	0.2271	20.2
10	5.01 - 10.0	1.3094	19.1
17	10.1 - 25.0	1.0298	6.0
6	25.1 - 50.0	1.1832	3.5
130	Overall	0.6113	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	47	0.004	0.0766
Digested Blank	46	0.004	0.0918

NITROGEN, TOTAL KJELDAHL (mg/L as N)
QUALITY CONTROL DATA FROM 10/01/99 TO 29/12/99
E3368



OXYGEN DEMAND, BIOCHEMICAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Before '61
Method Reference No.	E3182	Reporting Unit	mg/L as O
LIMS Product Code	BOD3182	Supervisor	P. Wilson
Sample Type/Matrix	Raw Sewage, Industrial Waste, Effluent, Drinking Water, Ground Water, Leachate, Surface Water		

SAMPLING:

Quantity Required:	400 mL
Container:	Glass or plastic

SAMPLE PREPARATION:

If necessary sample pH is adjusted to neutral and chlorine is removed by reaction with sodium sulphite.

ANALYTICAL PROCEDURE:

Oxygen depletion is measured as the difference in dissolved oxygen (DO) concentration. DO readings are taken prior to sample storage, and also at the end of storage in the dark at 20°C for five days (BOD₅). If necessary, dilutions are made with aerated, nutrient-enriched water to obtain a 25-75% oxygen depletion. If the sample has undergone any of the sample preparation steps listed above or if the sample is an industrial waste, a sewage seed is added. For such samples, calculation of an appropriate seed correction is required.

INSTRUMENTATION:

- YSI Model 59 DO meter (Yellow Springs Instrument Company) with DO probe equipped with stirrer and fitted with a Teflon membrane of 0.5 mil thickness which is permeable to oxygen (1 mil = 0.001 inch).
- Titration equipment for Winkler analysis of dissolved oxygen.
- Incubator (19-21°C); BOD bottles (300 mL)

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1
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CALIBRATION (DO):

The standard is air-saturated reversed osmosis deionized water. The DO content is read from a table (ORBISPHERE LABORATORIES - Pressure temperature dissolved oxygen table) after measuring its temperature and the barometric pressure in the laboratory.

OXYGEN DEMAND, BIOCHEMICAL cont'd

CONTROLS:

Calibration (DO)	2 QC solutions of Pure-DW water which have been partially stripped of DO by flushing with nitrogen. These "solutions", of different but unknown DO, are compared using the Oxygen meter and the Winkler titration procedure. The difference between the values for the two analytical methods is utilized as a slope control for the DO Analyzer.
Recovery (BOD5)*	3 Recovery standards prepared from a combination of Glucose and Glutamic Acid e.g. R1; the expected BOD5 is 67% of the oxygen requirement for complete oxidation.
Drift	Air saturated Pure-DW water after every 24 samples.
Blanks*	Pure-DW water and BOD dilution water

NOTES:

* These solutions are incubated for five days alongside samples.

OXYGEN DEMAND, BIOCHEMICAL (E3182)

QUALITY CONTROL DATA FROM 08/01/99 TO 29/12/99

Full Scale: to 9.0 mg/L as O at 20°C

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	86	0.00	-0.04	-0.04	0.09
B:	86	0.00	-0.01	-0.01	0.08

On any given day the calibration is accepted if the values obtained lie within the ranges:

-0.25 - 0.25

RECOVERIES:

Number of Data	Expected Depletion	Mean Depletion	Standard Deviation (1)
49	2.20	2.06	0.1357
49	4.34	4.23	0.1914
49	6.52	6.43	0.2806

DUPLICATES:

n Data Pairs	Sample Depletion Span	Standard Deviation (2)	Coefficient of variation(%)
40	0.0 - 1.8	0.2605	26.4
57	1.9 - 4.5	0.2648	8.4
51	4.6 - 9.0	0.3347	5.7
148	Overall	0.2897	

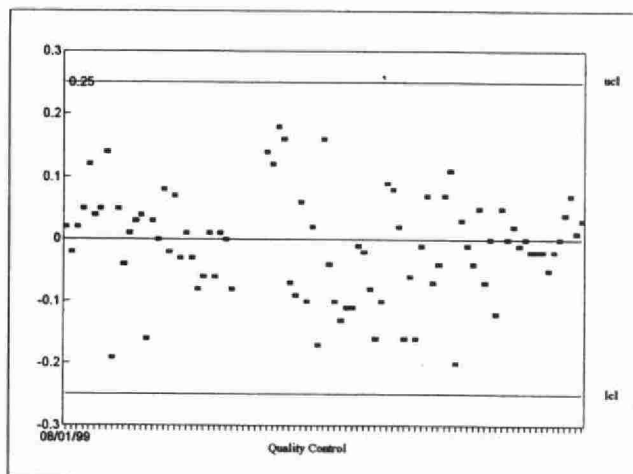
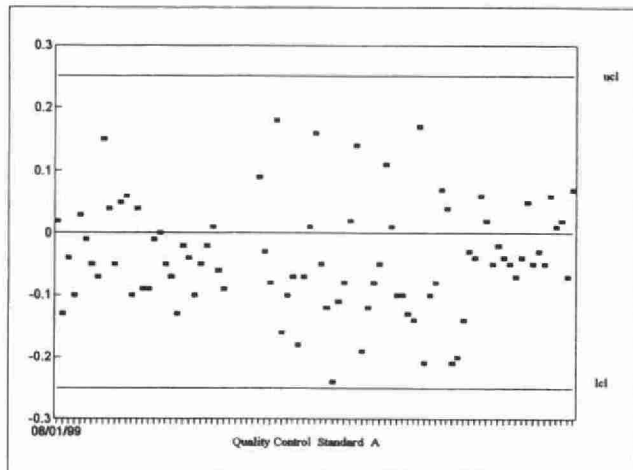
OTHER CHECKS:

	n	Mean	Standard Deviation (1)
5 Day Pure-DW Blank	49	0.139	0.1224
5 Day BOD Blank	49	0.145	0.1182

NOTES:

The final concentration of BOD in mg/L as O is determined by the oxygen depletion after 5 days at 20°C multiplied by a dilution and seed correction factor.

OXYGEN DEMAND, BIOCHEMICAL (mg/L as O)
QUALITY CONTROL DATA FROM 08/01/99 TO 29/12/99
E3182



OXYGEN DEMAND, CHEMICAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/07/82
Method Reference No.	E3170	Reporting Unit	mg/L as O
LIMS Product Code	COD3170	Supervisor	P. Wilson
Sample Type/Matrix	Drinking Water, Ground Water, Surface Water		

SAMPLING:

Quantity Required	25 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 149°C. Analysis is completed by automated colourimetric measurement of trivalent chromium. Approximate absorbance: 0.05 at the full scale level.

INSTRUMENTATION:

- Culture tubes with Teflon closures; mechanical-convection oven
- Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

REPORTING:

Maximum Significant Figures: 3	Current W value: 1	Current T value: 5
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CALIBRATION:

3 digested BL plus 3 digested standards

CONTROLS:

Calibration	2 digested standards, e.g. QCA
Drift	Undigested BL every 10 samples; standard plus BL at end of run
Recovery	2 digested standards, e.g. R1
Interference	Digested standard (40 mg/L as O) spiked with 50 mg/L Cl confirms suppression of chloride interference.

NOTES:

In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis is scheduled for completion within the week. The recovery standard is a material known to be very difficult to digest. The expected recovery is approximately 85%, based on long term experience. We continue to use this material in spite of the poor recovery, because if the slightest problem exists with the digestion step, the recovery falls off sharply to approximately 10%.

OXYGEN DEMAND, CHEMICAL (E3170)

QUALITY CONTROL DATA FROM 07/01/99 TO 15/12/99

Full Scale: to 40.0 mg/L as O

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	35	40.0	40.29	0.29	0.7585
B:	35	10.0	9.21	-0.79	0.8619
A+B:		50.0	49.59	-0.41	1.3026
A-B:		30.0	31.25	1.25	1.5035

s.d.(AB) S(between runs): 0.81 Sw(within run): 1.06 S/Sw: 0.76

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

46.3 - 53.7 for A+B
27.2 - 32.8 for A-B

RECOVERIES:

Number of Data	Expected Concentration	Mean Concentration	Standard Deviation (1)
35	40	37.17	1.5833
35	10	8.78	1.1112

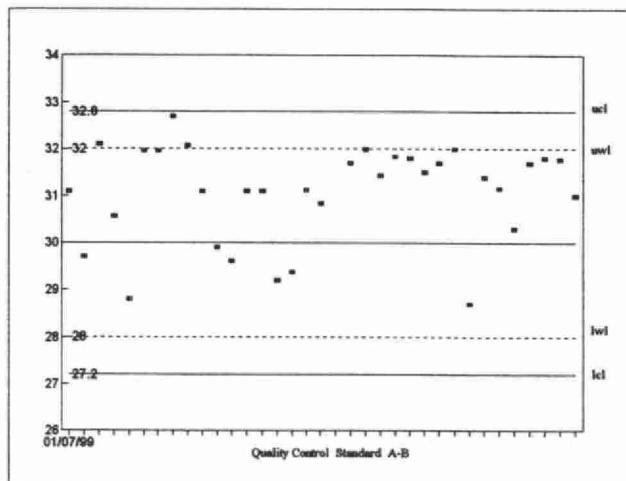
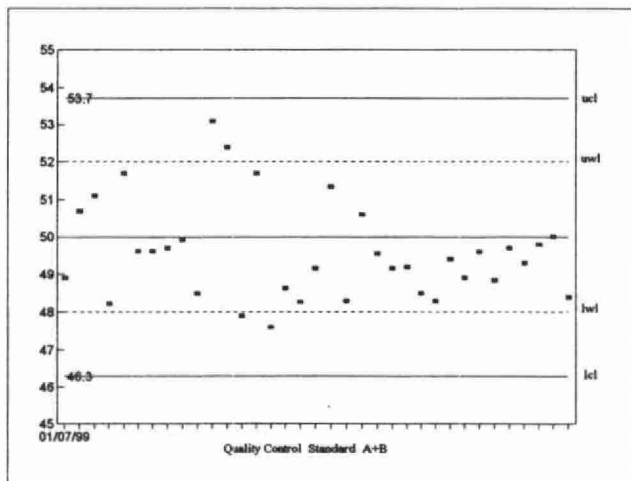
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
32	0 - 8	1.2768	24.2
37	9 - 20	1.4054	8.8
24	21 - 40	2.2927	8.5
93	Overall	1.6441	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Chloride Check	35	15.29	5.0443
Digested Blank	35	40.28	1.4813

OXYGEN DEMAND, CHEMICAL (mg/L as O)
QUALITY CONTROL DATA FROM 07/01/99 TO 15/12/99
E3170



OXYGEN DEMAND, CHEMICAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/07/82
Method Reference No.	E3246	Reporting Unit	mg/L as O
LIMS Product Code	COD3246	Supervisor	P. Wilson
Sample Type/Matrix	Raw Sewage, Industrial Waste, Ground Water, Leachate, Effluent, Sludge, Surface Water, Process Water		

SAMPLING:

Quantity Required	25 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 149°C. Analysis is completed by automated colourimetric measurement of trivalent chromium.

Approximate absorbance: 0.6 at the full scale level.

INSTRUMENTATION:

-Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

REPORTING:

Maximum Significant Figures: 3	Current W value: 2	Current T value: 10
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CALIBRATION:

2 digested BL plus 4 digested standards

CONTROLS:

Calibration	2 digested standards, e.g. QCA
Drift	Undigested BL every 10 samples; standard plus BL at end of run
Recovery	2 digested standards, e.g. R1
Interference	Digested standard (50 mg/L as O) spiked with 900 mg/L Cl confirms suppression of chloride interference.

NOTES:

In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis is scheduled for completion within the week. The recovery standard is a material known to be very difficult to digest. The expected recovery is approximately 85%, based on long term experience. We continue to use this material in spite of the poor recovery, because if the slightest problem exists with the digestion step, the recovery falls off sharply to approximately 10%.

OXYGEN DEMAND, CHEMICAL (E3246)

QUALITY CONTROL DATA FROM 07/01/99 TO 22/12/99

Full Scale: to 400.0 mg/L as O

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	23	400	397.0	-3.0	4.8053
B:	23	100	99.3	-0.7	3.5373
A+B:		500	496.3	-3.7	6.7455
A-B:		300	297.7	-2.3	5.0701

s.d.(AB) S(between runs): 4.2 Sw(within run): 3.6 S/Sw: 1.2

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

477.5 - 522.5 for A+B
285.0 - 315.0 for A-B

RECOVERIES:

Number of Data	Expected Concentration	Mean Concentration	Standard Deviation (1)
23	390	393.6	5.5437
23	98	91.3	4.9203

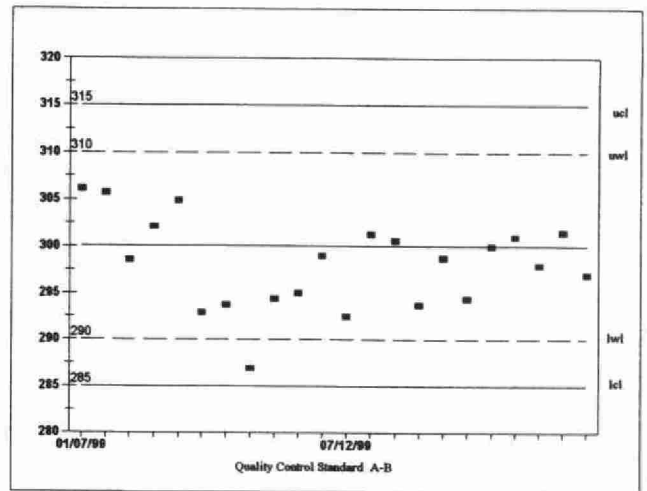
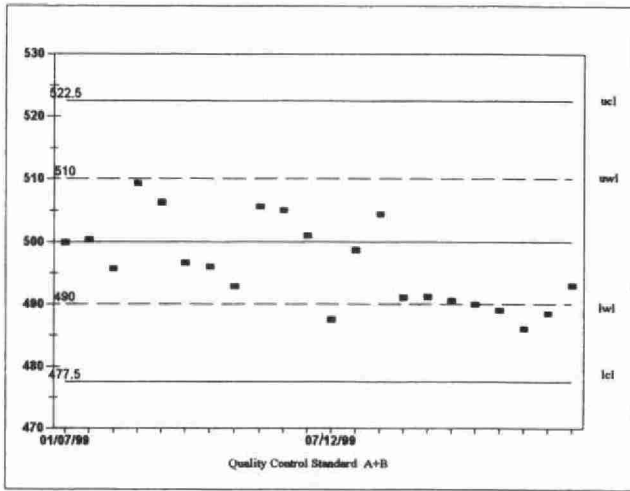
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
24	0 - 80	2.7103	7.0
10	81 - 200	4.4151	3.1
7	201 - 400	5.4346	1.8
41	Overall	3.7546	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Chloride Check	23	56.0	8.4989
Digested Blank	23	28.0	5.5437

OXYGEN, DEMAND CHEMICAL (mg/L as O)
QUALITY CONTROL DATA FROM 01/07/99 TO 12/22/99
E3246



pH

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	01/01/76
Method Reference No.	E3042	Reporting Units	dimensionless
LIMS Product Code	PHALK3042	Manager	W. Scheider
Sample Type/Matrix:	Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required:	150 mL
Container:	250 mL Amber polyethylene or BOD bottle filled to the brim; screw caps with cone-shaped liners are preferred.

ANALYTICAL PROCEDURE:

pH is measured directly on a stirred sample (50 mL) at room temperature. Stirring rate, beaker size, degree of electrode immersion and room temperature range are uniform for all samples and standards. Alkalinity (Gran) and Alkalinity, Total Fixed End Point are performed simultaneously.

INSTRUMENTATION:

Auto Burette, Auto Sampler, pH Meter all controlled by a PC, and combined glass electrode.

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7

CONTROLS:

Calibration	BL plus 2 standards, e.g. QCA
Drift	2 standard buffers - 2 times daily

pH (E3042)

QUALITY CONTROL DATA FROM 08/01/99 TO 17/12/99

Analytical Range: to 14.00 Dimensionless

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	68	6.86	6.856	-0.004	0.0167
B:	68	4.01	4.03	0.02	0.0350
A+B:		10.87	10.89	0.02	0.0381
A-B:		2.85	2.82	-0.03	0.0395

s.d.(AB) S(between runs): 0.0274 Sw:(within run): 0.0279 S/Sw: 0.98

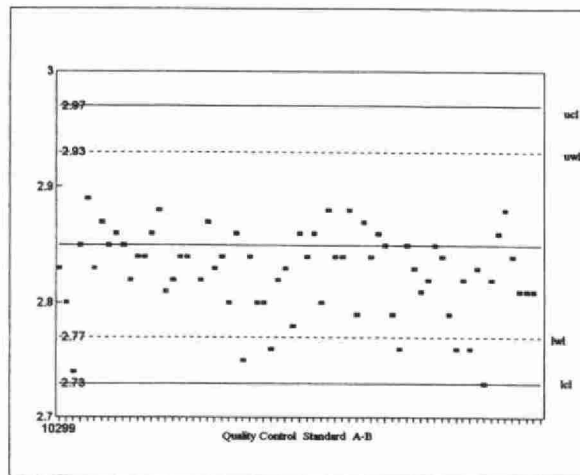
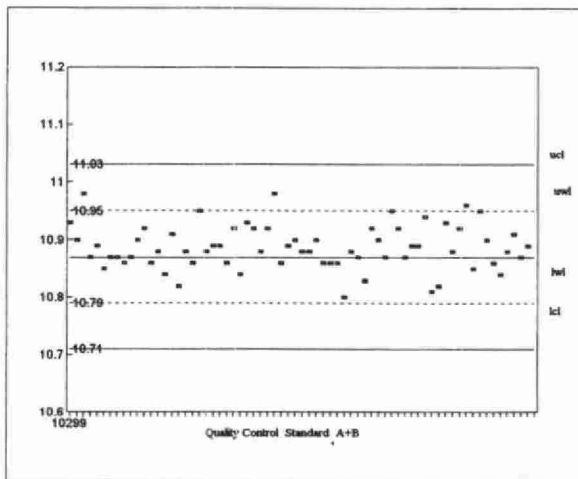
On any given day the calibration is accepted if the values obtained lie within the ranges:

10.65 - 11.07 for A+B
2.72 - 3.00 for A-B

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
65	3.5 - 5.00	0.0224	0.5
64	5.01 - 6.00	0.0287	0.5
53	6.01 - 7.00	0.0369	0.6
7	7.01 - 9.00	0.0866	1.1
189	Overall	0.0333	

pH
 QUALITY CONTROL DATA FROM 08/01/99 TO 17/12/99
 E3042



pH

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	09/07/80
Method Reference No	E3218	Reporting Units	Dimensionless
LIMS Product Code	PHALCO3218, CONDPH3218	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Effluent, Industrial Waste, Raw Sewage, Drinking Water, Ground Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or Plastic

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (20.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards. Total fixed endpoint alkalinity, and conductivity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration	2 QC standards e.g. QCA
Drift	In run standards throughout the run (diluted tap water 50% V/V)

pH (E3218)

QUALITY CONTROL DATA FROM 05/01/99 TO 29/12/99

Analytical Range: to 14.00 Dimensionless

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	54	7.41	7.43	0.02	0.0174
B:	54	4.45	4.48	0.03	0.0532
A+B:		11.86	11.91	0.05	0.0550
A-B:		2.96	2.95	-0.01	0.0569

s.d.(AB) S(between runs): 0.040 Sw(within run): 0.040 S/Sw: 1.0

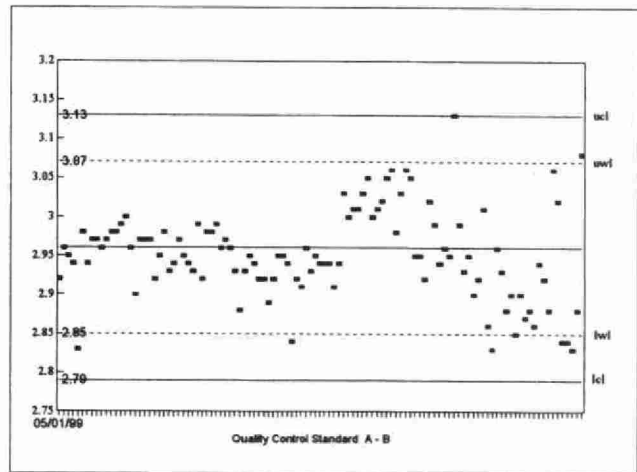
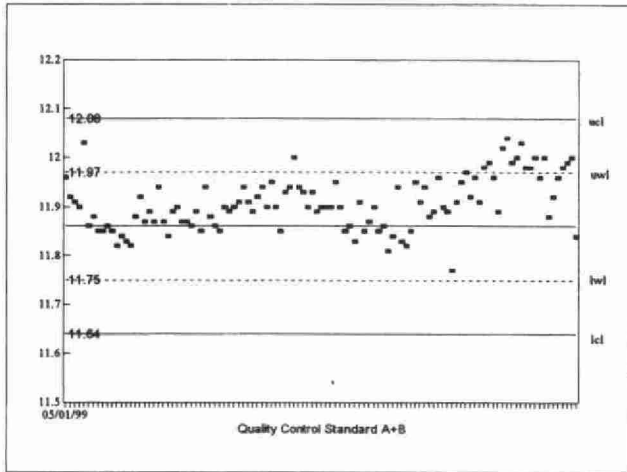
On any given day the calibration is accepted if the values obtained lie within the ranges:

11.64 - 12.08 for A+B
2.79 - 3.13 for A-B

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
37	1.00 - 7.00	0.0222	0.4
169	7.01 - 8.00	0.0226	0.4
112	8.01 - 12.00	0.0249	0.3
318	Overall	0.0234	

pH
QUALITY CONTROL DATA FROM 05/01/99 TO 29/12/99
E3218



pH

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/05/79
Method Reference No	E3248	Reporting Units	Dimensionless
LIMS Product Code	PHACD3248,PH3248	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Raw Sewage, Drinking Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	15 mL
Container	Glass or Plastic

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (15.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards.

Total fixed endpoint acidity and Gran acidity can be determined simultaneously if the sample volume exceeds 50 mL .

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
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NOTES:

A new automated system was introduced in Sept' 96.

pH (E3248)

QUALITY CONTROL DATA FROM 02/02/99 TO 07/12/99

Analytical Range: to 14.00 Dimensionless

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	17	4.45	4.416	-0.034	0.0563
B:	17	3.75	3.700	-0.050	0.0320
A+B:		8.20	8.116	-0.084	0.0789
A-B:		0.70	0.716	0.016	0.0466

s.d.(AB) S(between runs): 0.032

Sw:(within run): 0.023

S/Sw: 1.4

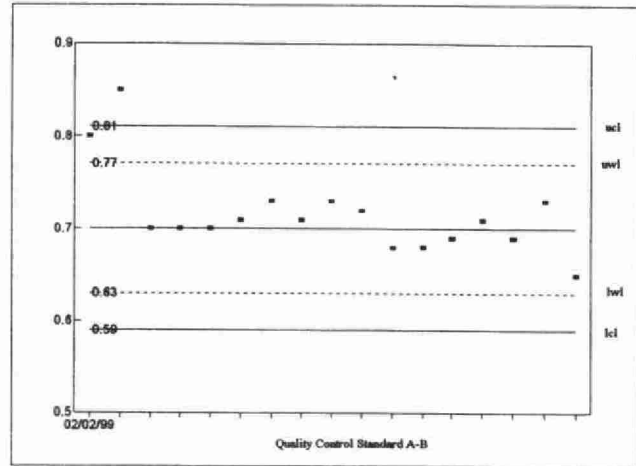
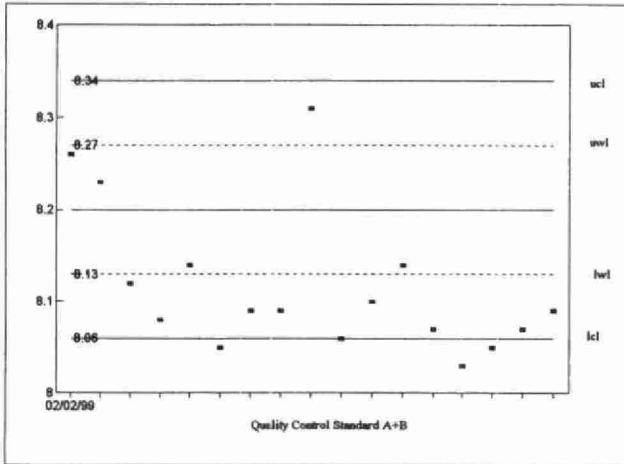
On any given day the calibration is accepted if the values obtained lie within the ranges:

8.06 - 8.34 for A+B
0.59 - 0.81 for A-B

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
2	2.50 - 4.00	0.0100	0.3
23	4.01 - 5.00	0.0189	0.4
11	5.01 - 8.00	0.1151	1.8
36	Overall	0.0664	

pH
 QUALITY CONTROL DATA FROM 02/02/99 TO 07/12/99
 E3248



PHENOLICS, REACTIVE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/74
Method Reference No.	E3179	Reporting Unit	µg/L as Phenol
LIMS Product Code	PHEN3179	Supervisor	P.Wilson
Sample Type/Matrix	Ground Water, Surface Water, Effluent, Drinking Water, Leachate, Raw Sewage, Industrial Waste, Process Water, Precipitation		

SAMPLING:

Quantity Required	250 mL
Container	Glass, (Phenol bottle with white cap containing preservative is available)
Preservative	Sulfuric acid to pH 1.5 - 2

ANALYTICAL PROCEDURE:

Samples are automatically distilled from an acid media, and reactive phenolics in the distillate are determined colourimetrically by formation of an antipyrene dye through reactions with 4-aminoantipyrene and potassium ferricyanide.

Approximate absorbance: 0.03 at the full scale level.

INSTRUMENTATION:

Basic automated modular continuous flow system plus a distillation module. Colourimetric measurement is through a 5.0 cm. light path at 505 nm.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1
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CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA (see note)
Drift	BL ,standard ,BL every 10 samples

NOTES:

An additional Quality Control Standard (QCC) was added to the method in March 1997.

PHENOLICS, REACTIVE

QUALITY CONTROL DATA FROM 19/01/99 TO 12/12/99

Laboratory Unit: Colourimetry

Full Scale: to 50.0 µg/L as Phenol

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	41	40	40.05	0.05	0.4346
B:	41	10	10.28	0.28	0.1664
C:	41	5	5.16	0.16	0.1577
A+B:		50	50.32	0.32	0.5126
A-B:		30	29.76	-0.24	0.4128
B+C:		15	15.43	0.43	0.2825
B-C:		5	5.12	0.12	0.1672

s.d.(AB)

S(between runs):0.33

Sw(within run): 0.30

S/Sw: 1.1

s.d.(BC)

S(between runs):0.16

Sw(within run): 0.12

S/Sw: 1.4

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

47.8	-	52.2	for	A+B
28.3	-	31.7	for	A-B
14.0	-	16.0	for	B+C
4.25	-	5.75	for	B-C

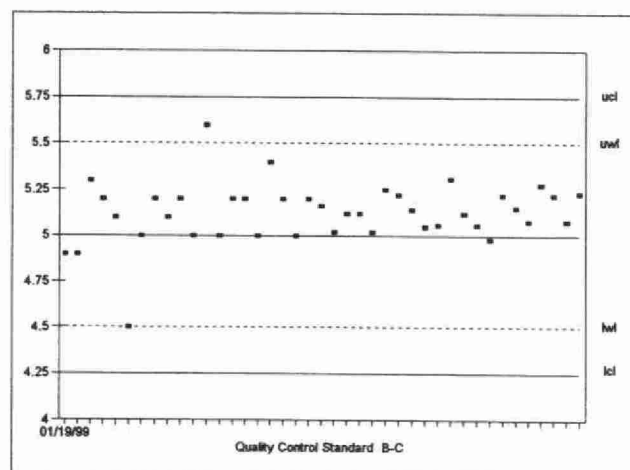
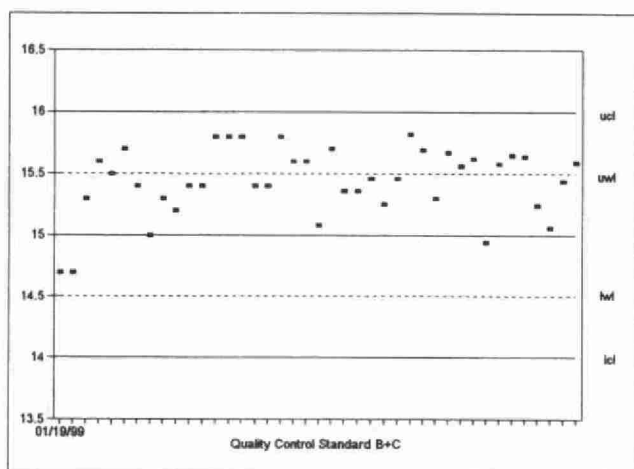
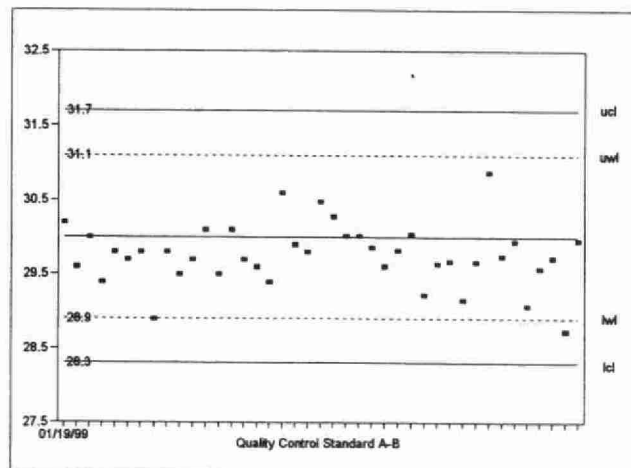
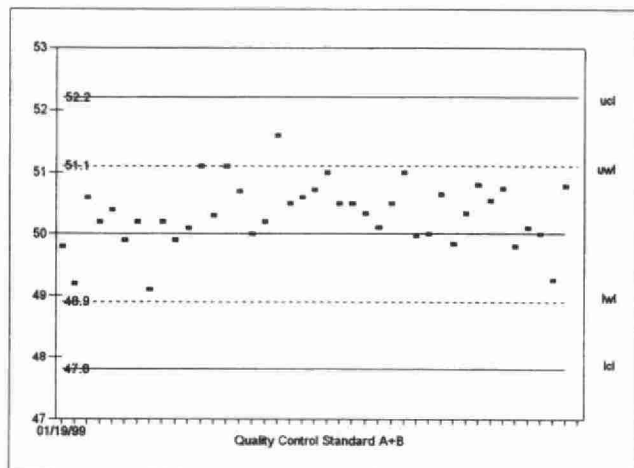
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
102	0.00 - 5.0	0.1119	22.3
5	5.1 - 10.0	0.2169	2.9
5	10.1 - 25.0	0.2169	2.9
5	25.1 - 50.0	0.3415	0.9
117	Overall	0.1527	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	40	0.115	0.0921

PHENOLIC, REACTIVE ($\mu\text{g/L}$ as Phenol)
QUALITY CONTROL DATA FROM 19/01/99 TO 12/12/99
E3179



PHOSPHORUS, REACTIVE ortho-PHOSPHATE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/79
Method Reference No.	E3364	Reporting Unit	mg/L as P
LIMS Product Code	DISNUT3364	Supervisor	P.Wilson
Sample Type/Matrix	Dried Sludge, Sediment, Soil, Vegetation, Drinking Water, Ground Water, Surface Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Ortho-phosphate is determined on the supernatant of a settled sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.2 at the full scale level.

Ammonia plus ammonium, nitrite, and nitrate plus nitrite are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using IR sensitive phototube.

Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.0005	Current T value: 0.0025
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL ,standard ,and BL after every 10 samples

NOTES:

The HP data capture / processing system was replaced by Labtronics in August 1999.

PHOSPHORUS, REACTIVE ortho-PHOSPHATE (E3364)

QUALITY CONTROL DATA FROM 04/01/99 TO 23/12/99

Laboratory Unit: Colourimetry

Full Scale: to 0.100 mg/L as P

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	106	0.080	0.07996	-0.00004	0.0014
B:	106	0.040	0.0405	0.0005	0.0009
C:	106	0.008	0.008	0.0001	0.0007
A+B:		0.120	0.1205	0.0005	0.0016
A-B:		0.040	0.0394	-0.0006	0.0018
B+C:		0.048	0.0486	0.0006	0.0014
B-C:		0.032	0.0324	0.0004	0.0010

s.d.(AB) S(between runs):0.0012

s.d.(BC) S(between runs):0.0008

Sw(within run): 0.0013

Sw(within run): 0.0007

S/Sw: 1.0

S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.1152 - 0.1248 for A+B

0.0364 - 0.0436 for A-B

0.0448 - 0.0512 for B+C

0.0296 - 0.0344 for B-C

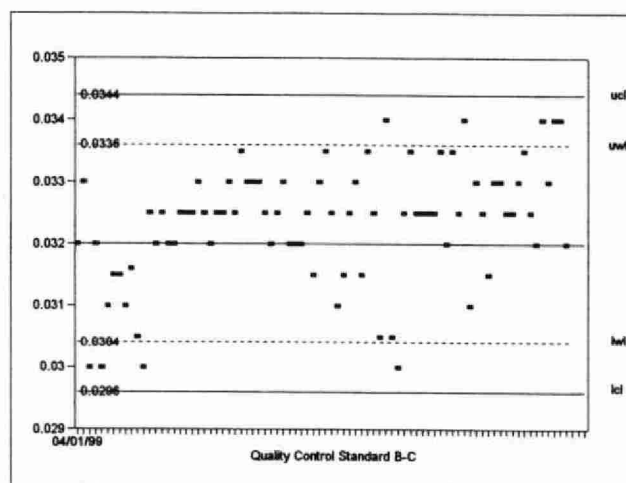
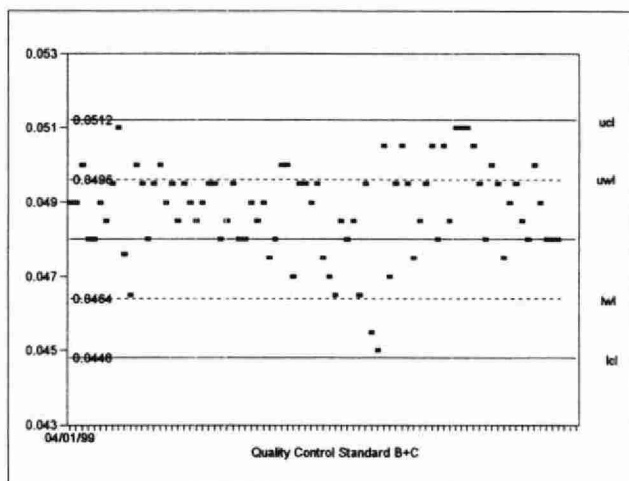
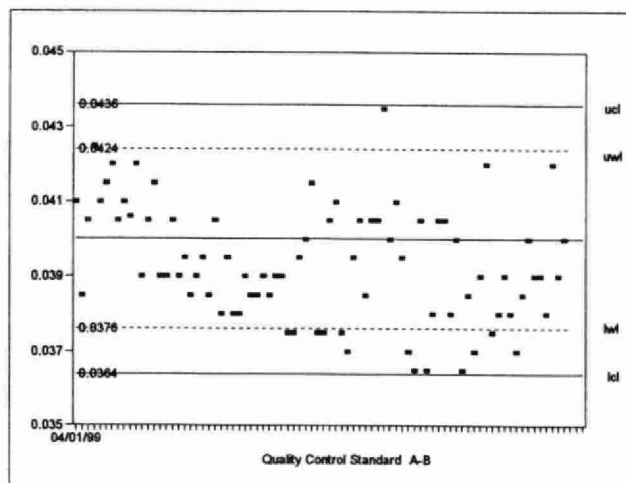
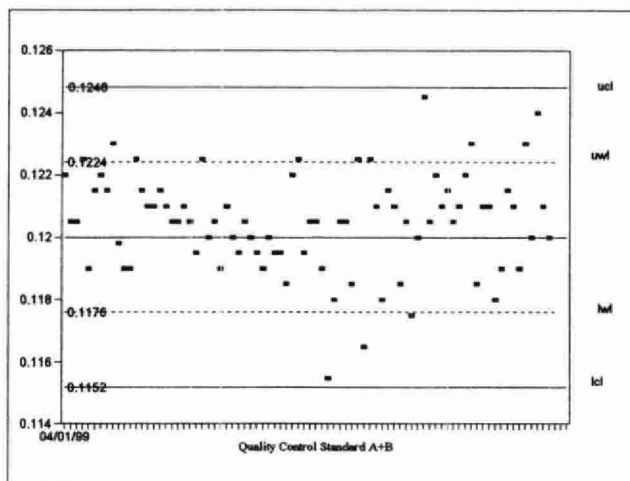
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
221	0 - 0.010	0.0011	29.6
34	0.011 - 0.020	0.0021	14.8
36	0.021 - 0.050	0.0050	16.0
11	0.051 - 0.100	0.0021	3.1
302	Overall	0.0021	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	106	0.0004	0.0018

PHOSPHORUS, REACTIVE ortho-PHOSPHATE (mg/L as P)
 QUALITY CONTROL DATA FROM 04/01/99 To 23/12/99
 E3364



PHOSPHORUS, REACTIVE ortho-PHOSPHATE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/79
Method Reference No	E3366	Reporting Unit	mg/L as P
LIMS Product Code	DISNUT3366	Supervisor	P.Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Leachate, Drinking Water, Ground Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Ortho-phosphate is determined on the supernatant of a settled sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.5 at the full scale level.

Ammonia plus ammonium, nitrite, and nitrate plus nitrite are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using IR sensitive phototube.

Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.02	Current T value: 0.1
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL ,standard and BL every 10 samples

NOTES:

The HP capture / processing system was replaced by Labtronics in October 1999.

PHOSPHORUS, REACTIVE ortho-PHOSPHATE (E3366)

QUALITY CONTROL DATA FROM 05/01/99 TO 23/12/99

Laboratory Unit: Colourimetry

Full Scale: to 10.0 mg/L as P

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	93	8.00	7.980	-0.020	0.0817
B:	93	4.00	4.014	0.014	0.0416
C:	93	0.800	0.820	0.020	0.0226
A+B:		12.0	11.994	-0.006	0.0930
A-B:		4.00	3.965	-0.035	0.0904
B+C:		4.80	4.834	0.034	0.0514
B-C:		3.20	3.194	-0.006	0.0428

s.d.(AB) S(between runs):0.064

Sw(within run): 0.064

S/Sw: 1.0

s.d.(BC) S(between runs):0.033

Sw(within run): 0.030

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

11.71	-	12.29	for	A+B
3.78	-	4.22	for	A-B
4.66	-	4.94	for	B+C
3.09	-	3.31	for	B-C

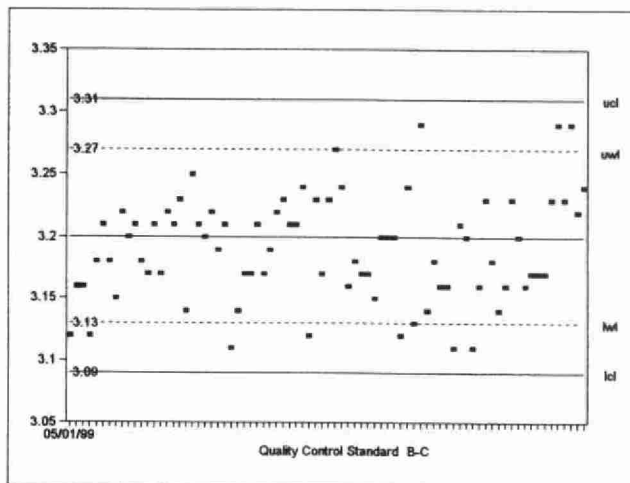
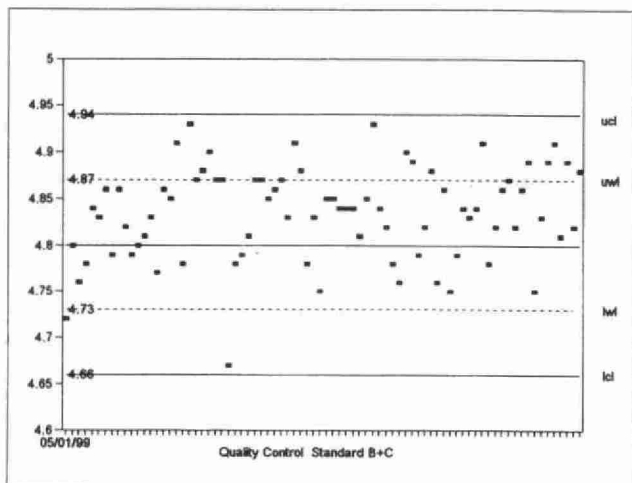
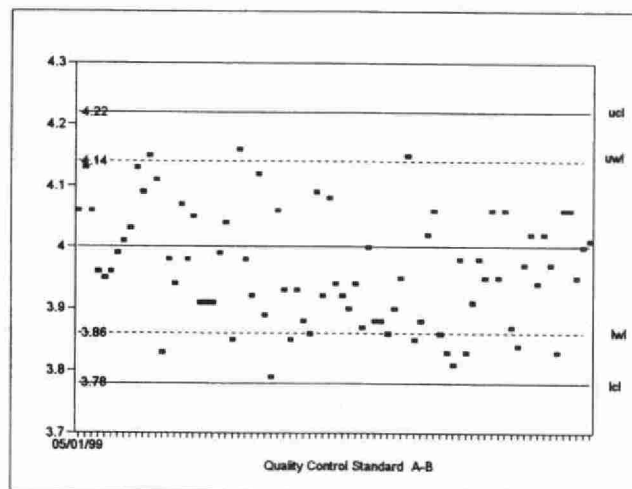
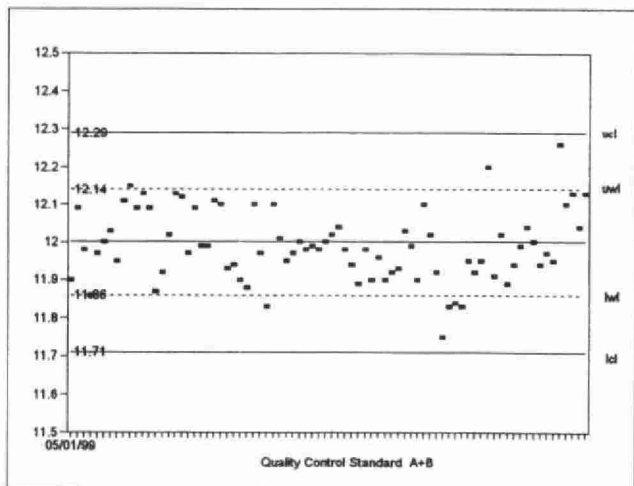
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
238	0 - 1.00	0.0386	32.0
14	1.01 - 2.00	0.0530	3.6
18	2.01 - 5.00	0.0465	1.5
27	5.01 - 10.0	0.0562	1.3
297	Overall	0.0414	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	93	0.0084	0.0418

PHOSPHORUS, REACTIVE ortho-PHOSPHATE (mg/L as P)
 QUALITY CONTROL DATA FROM 05/01/99 TO 23/12/99
 E3366



PHOSPHORUS, TOTAL

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	22/03/79
Method Reference No.	E3036	Reporting Unit	µg/L as P
LIMS Product Code	TP3036	Manager	F.Tomassini
Sample Type/Matrix	Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required:	35 mL
Container:	Specially marked Pyrex culture tubes with Teflon-lined caps

ANALYTICAL PROCEDURE:

Digestion reagent is added and samples are autoclaved in sulphuric acid-potassium persulphate media at 121°C for 60 min. The orthophosphate content of the digestate is determined colourimetrically by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.3 at the full scale level

INSTRUMENTATION:

Autoclave plus basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1
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CALIBRATION:

BL plus 9 undigested standards

CONTROLS:

Calibration	LTBL plus 3 undigested standards, e.g. QCA
Drift	BL every 10 samples and BL plus 1 undigested standard every 20 samples
Recovery	3 digested BL plus 3 digested standards, e.g. R1

NOTES:

System is calibrated with undigested standards, but sample concentrations are corrected to reflect the run's value for the digested blank.

PHOSPHORUS, TOTAL (E3036)

QUALITY CONTROL DATA FROM 14/01/99 TO 07/12/99

Analytical Range: to 50 µg/L

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	29	45.0	46.6	1.6	1.3660
B:	29	13.5	13.49	-0.01	0.4447
C:	29	4.5	4.0	-0.5	0.8515
A+B:		58.5	60.0	1.5	1.4207
A-B:		31.5	33.0	1.5	1.4525
B+C:		18.0	17.6	-0.4	0.6669
B-C:		9.0	9.4	0.4	0.4780

s.d.(AB) S(between runs): 1.01 Sw(within run): 1.03 S/Sw: 0.99
s.d.(BC) S(between runs): 0.68 Sw(within run): 0.34 S/Sw: 2.00

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

53.5 - 63.5 for A+B
27.5 - 35.5 for A-B
16.0 - 20.0 for B+C
7.5 - 10.5 for B-C

RECOVERIES:

Number of Data	Expected Concentration	Mean Concentration	Standard Deviation (1)
29	35	35.3	2.3804
29	14	14.8	0.6427
29	7	7.3	0.6631

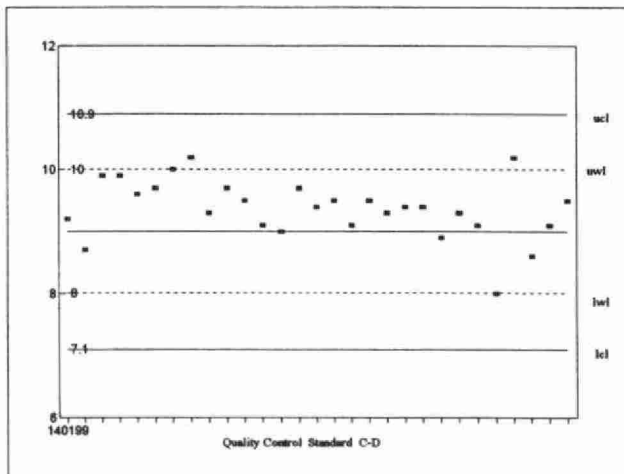
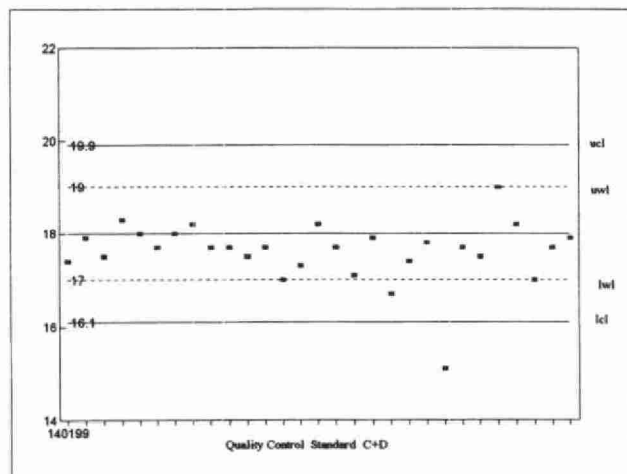
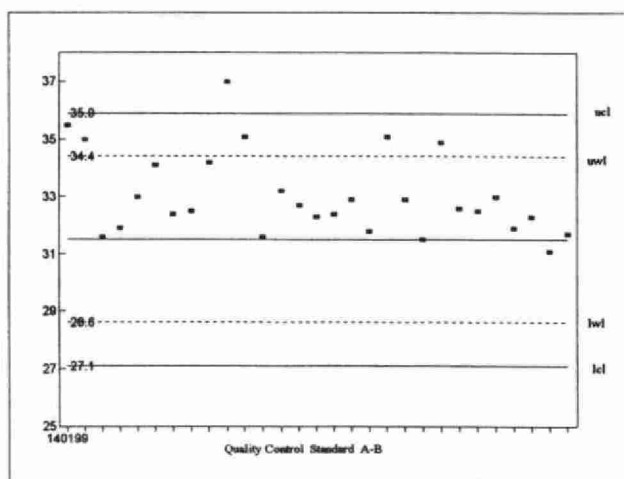
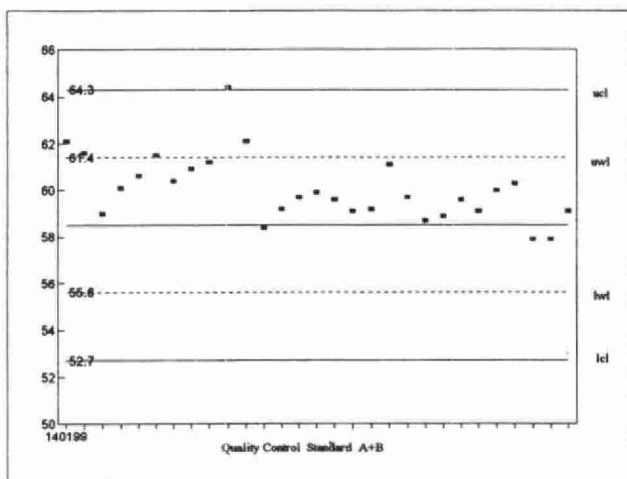
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
38	0.0 - 10.0	0.3636	5.9
22	10.1 - 20.0	0.8096	5.4
17	20.1 - 50.0	0.7583	2.5
3	50.1 - 100.0	1.2254	4.1
80	Overall	0.6843	

OTHER CHECKS:

	n	Data Mean	Standard (1) Deviation
Long Term Blank	29	-0.172	0.3326
Digested Blank	29	1.255	0.6620

PHOSPHORUS , TOTAL (ug/L as P)
QUALITY CONTROL DATA FROM 14/01/99 TO 07/12/99
E3036



PHOSPHORUS, TOTAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Mar '89
Method Reference No.	E3116	Reporting Unit	mg/g as P
LIMS Product Code	TNP3116	Supervisor	P. Wilson
Sample Type/Matrix	Soil, Sediment, Dried Sludge		

SAMPLING:

Quantity Required	0.08 to 0.4 g
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Phosphorus compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is filtered and the filtrate is analyzed using an automated colourimetric system.

INSTRUMENTATION:

Hot plate

Basic automated modular continuous flow system : Colourimetric measurement is through a 5 cm. light path at 660 nm.

Data capture, and processing via a microcomputer system

REPORTING:

Maximum Significant Figures: 2 decimal places	Current W value: 0.02	Current T value: 0.10
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CALIBRATION:

3 High and 2 Low Calibration Standards

CONTROLS:

Drift	4 BL's per run; high and low calibration standard at the end of the run
Recovery	2 digested standards (R1,R2) to monitor digestion procedure, a non certified reference material RSM2781 to monitor digestion procedure for sludge samples and an in house composite (RS92) to monitor digestion procedure for sediment and soils.

NOTES:

System is calibrated with undigested standards.

Recovery Standards R1 and R2 are standards prepared in Pure Water and are less complex than soil and sludge matrices. R1 and R2 showed levels within limits for 1999 with 2 exceptions for R1 (Apr 7, Nov 17) at 5% and one exception for R2 (Aug18) at -14%. RS92 showed the lower recovery limit was exceeded by -29% and the RSM2781 showed both low and high recoveries -14 to 34%. The digestion procedure is still under investigation.

PHOSPHORUS, TOTAL (E3116)

QUALITY CONTROL DATA FROM 07/04/99 TO 03/12/99

Full Scale: 2 mg/g as P

QUALITY CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
RS92 In House Soil Composite	34	0.47	0.44	-0.03	0.0493
RSM-2781 -Reference Standard Material	34	25.5	24.2	-1.3	2.8903

The run is accepted if the control values obtained lie within the ranges:

0.40 - 0.54 for RS92
23.3 - 25.1 for RSM-2781

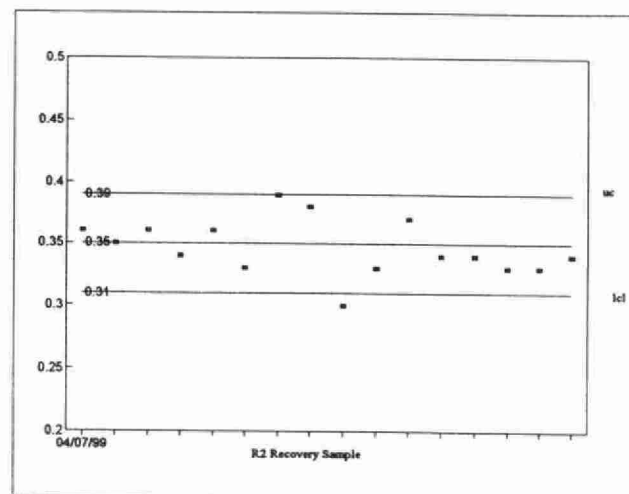
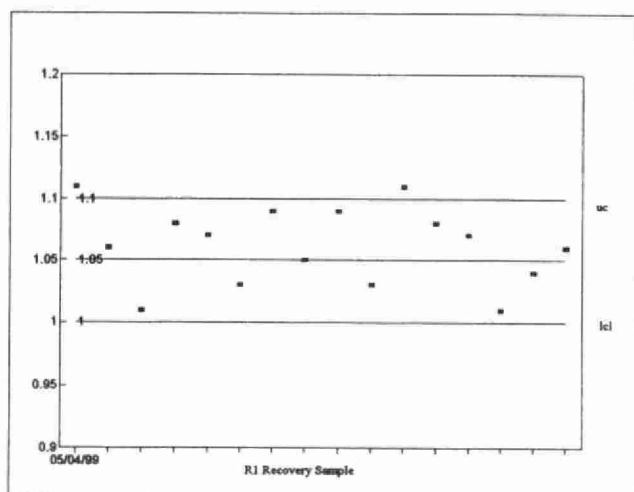
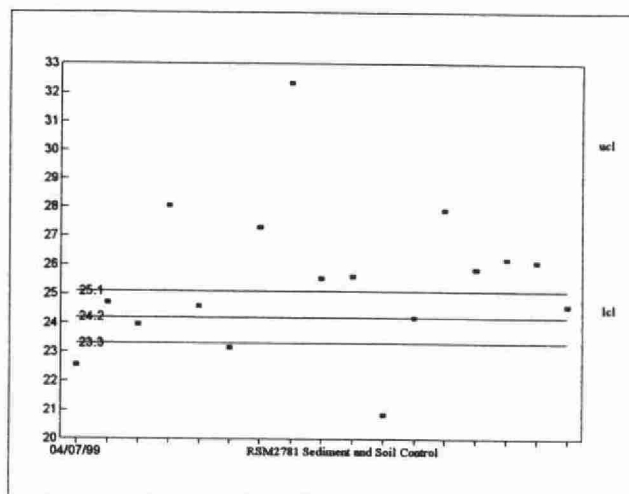
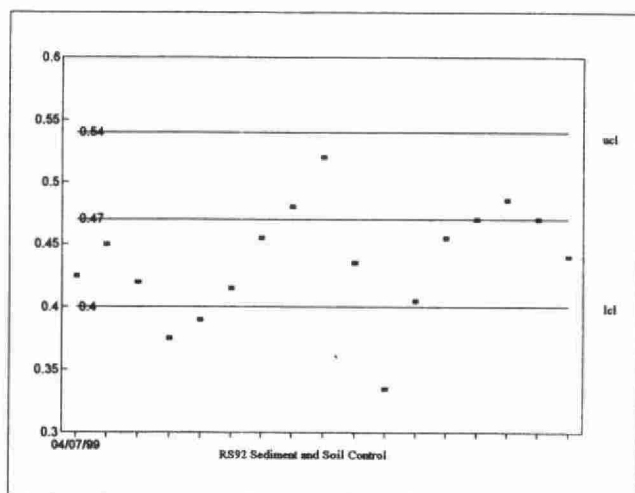
Recovery Standards

	n	Expected Concentration	Mean Concentration	Standard Deviation (1)
R1	16	1.05	1.062	0.0317
R2	16	0.35	0.347	0.0224

DUPLICATES: (Sediment/Soils)

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
35	0.00 - 0.50	0.1307	17.3
7	0.51 - 1.00	0.0635	2.1
2	1.00 - 2.50	0.1278	2.3
0	2.51 - 5.00	N.A.	N.A.
44	Overall	0.1223	

PHOSPHORUS , TOTAL (mg/L as P)
QUALITY CONTROL DATA FROM 07/04/99 TO 03/12/99
E3116



PHOSPHORUS, TOTAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Mar '89
Method Reference No.	E3118	Reporting Unit	mg/g as P
LIMS Product Code	TNP3118	Supervisor	J. McBride
Sample Type/Matrix	Vegetation, Moss Bag		

SAMPLING:

Quantity Required	0.02 to 0.04 g
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Phosphorus compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is analyzed using an automated colourimetric system.

INSTRUMENTATION:

Hot plate.

Basic automated modular continuous flow system : Colourimetric measurement is through a 5 cm. light path at 660 nm.

Data capture, and processing via a microcomputer system.

REPORTING:

Maximum Significant Figures: 2 decimal places	Current W value: 0.02	Current T value: 0.10
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CALIBRATION:

3 High and 2 Low Calibration Standards

CONTROLS:

Calibration	In house composite A-VEG, plus QC VEG (Pine Needles)
Drift	4 BL's per run; high and low calibration standard at the end of the run
Recovery	1 digested BL plus 4 digested standards

NOTES:

System is calibrated with undigested standards.

There is not enough data to report QC for 1999.

PHOSPHORUS, TOTAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/79
Method Reference No	E3367	Reporting Unit	mg/L as P
LIMS Product Code	TOTNUT3367	Supervisor	P.Wilson
Sample Type/Matrix	Precipitation, Drinking Water, Ground Water , Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digesters kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.4 at the full scale level.

Total Kjeldahl nitrogen is determined simultaneously.

INSTRUMENTATION:

Three Block digesters

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.

Data capture, reduction, and processing via a multi-stage microcomputer system

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.002	Current T value: 0.01
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CALIBRATION:

BL plus 7 undigested standards

CONTROLS:

Calibration	LTBL plus 3 undigested standards, e.g. QCA
Drift	BL , undigested standard , BL every 10 samples
Recovery	3 digested BL plus 3 digested standards in duplicate, e.g. R1

NOTE:

The HP capture / processing system was replaced by Labtronics in May 1999

PHOSPHORUS, TOTAL (E3367)

QUALITY CONTROL DATA FROM 11/01/99 TO 16/12/99

Full Scale: to 0.200 mg/L as P

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	104	0.160	0.1605	0.0005	0.0012
B:	104	0.080	0.0807	0.0007	0.0008
C:	104	0.016	0.0159	-0.0001	0.0009
A+B:		0.240	0.2412	0.0012	0.0017
A-B:		0.080	0.0798	-0.0002	0.0013
B+C:		0.096	0.0966	0.0006	0.0014
B-C:		0.064	0.0649	0.0009	0.0011

s.d.(AB) S(between runs): 0.0011 Sw(within run): 0.0009 S/Sw: 1.2
s.d.(BC) S(between runs): 0.0009 Sw(within run): 0.0008 S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.2332 - 0.2468 for A+B
0.0749 - 0.0851 for A-B
0.092 - 0.100 for B+C
0.061 - 0.067 for B-C

RECOVERIES:

Number of Data	Expected Concentration	Mean Concentration	Standard Deviation (1)
100	0.140	0.1393	0.0055
100	0.084	0.0838	0.0043
100	0.028	0.0281	0.0025

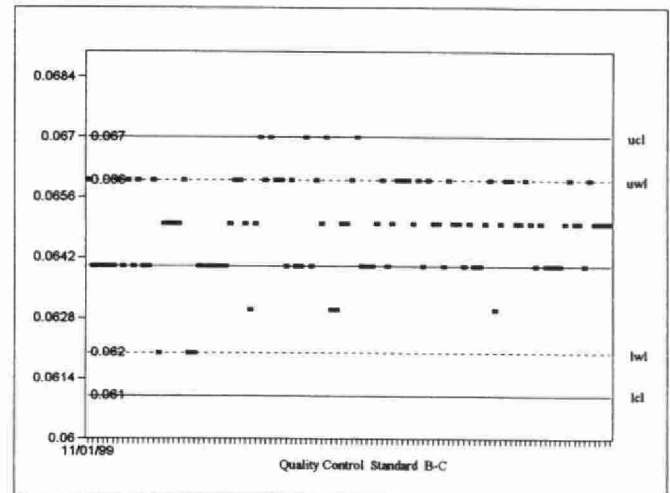
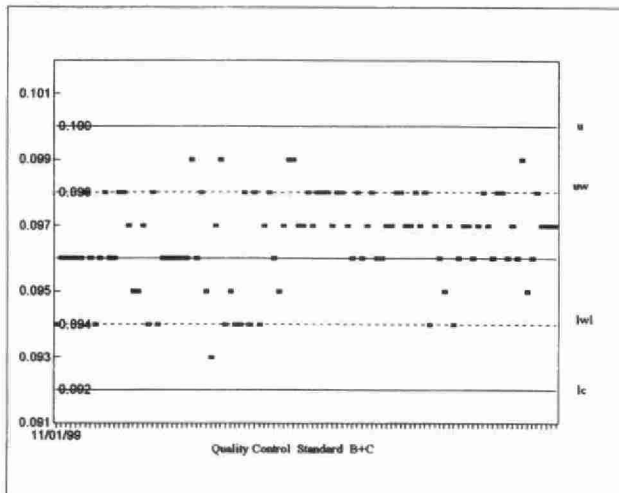
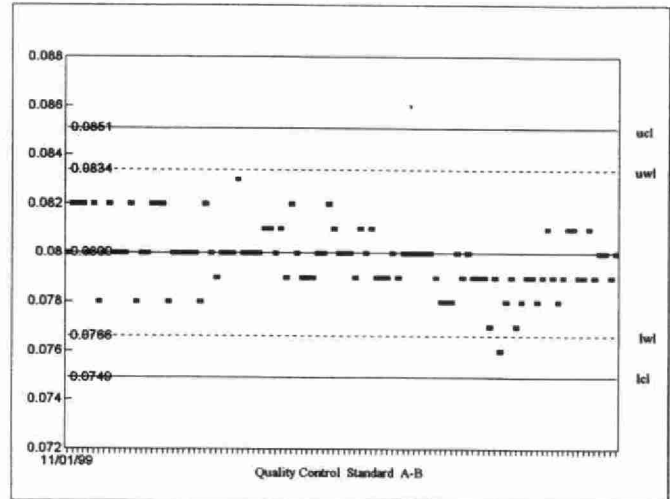
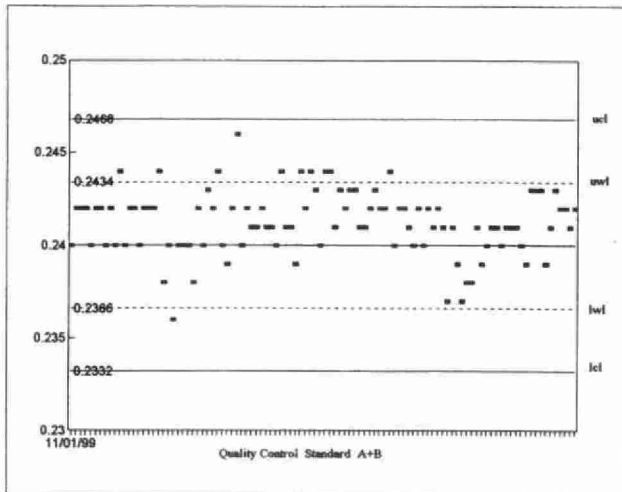
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
237	0 - 0.020	0.0045	52.6
24	0.021 - 0.040	0.0071	24.7
31	0.041 - 0.100	0.0075	12.3
4	0.101 - 0.200	0.0330	27.0
296	Overall	0.0064	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	104	0.0004	0.0014
Digested Blank	100	0.0008	0.0019

PHOSPHORUS, TOTAL (mg/L as P)
QUALITY CONTROL DATA FROM 11/01/99 TO 16/12/99
E3367



PHOSPHORUS, TOTAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/79
Method Reference No.	E3368	Reporting Unit	mg/L as P
LIMS Product Code	TOTNUT3368	Supervisor	J. McBride
Sample Type/Matrix	Sludge, Raw Sewage, Industrial Waste, Drinking Water, Effluent, Ground Water, Process Water, Leachate		

SAMPLING:

Quantity Required	50 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digestors kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.8 at the full scale level.

Total Kjeldahl Nitrogen is determined simultaneously.

INSTRUMENTATION:

3-Block digesters

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using an IR sensitive phototube. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.02	Current T value: 0.10
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL every 10 samples; undigested standard every 20 samples
Recovery	3 digested BL plus 3 digested standards in duplicate, e.g. R1

NOTES:

System is calibrated with undigested standards.

The HP capture / processing system was replaced by Labtronics in April 1999

PHOSPHORUS, TOTAL (E3368)

QUALITY CONTROL DATA FROM 08/01/99 TO 29/12/99

Full Scale: to 10.0 mg/L as P

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	51	8.0	8.001	0.001	0.0339
B:	51	4.0	4.017	0.017	0.0238
C:	51	0.8	0.808	0.008	0.0099
A+B:		12.0	12.022	0.022	0.0451
A-B:		4.0	3.988	-0.012	0.0374
B+C:		4.8	4.826	0.026	0.0246
B-C:		3.2	3.209	0.009	0.0270

s.d.(AB) S(between runs): 0.0293

Sw(within run): 0.0264

S/Sw: 1.1

s.d.(BC) S(between runs): 0.0183

Sw(within run): 0.0191

S/Sw: 0.96

The calibration is accepted if the calibration control values obtained lie within the ranges:

11.87 - 12.13 for A+B
 3.903 - 4.097 for A-B
 4.732 - 4.868 for B+C
 3.149 - 3.251 for B-C

RECOVERIES:

Number of Data	Expected Concentration	Mean Concentration	Standard Deviation (1)
51	7	6.91	0.2124
51	4.2	4.13	0.2579
51	1.4	1.40	0.0501

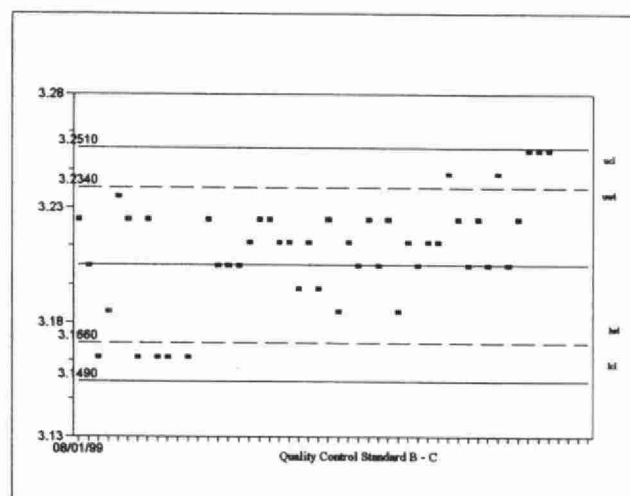
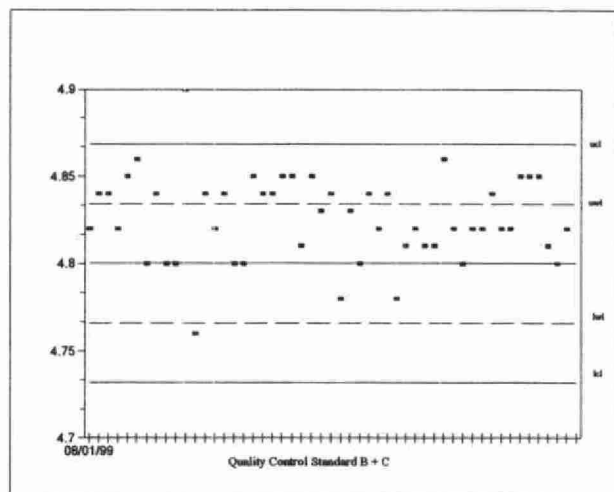
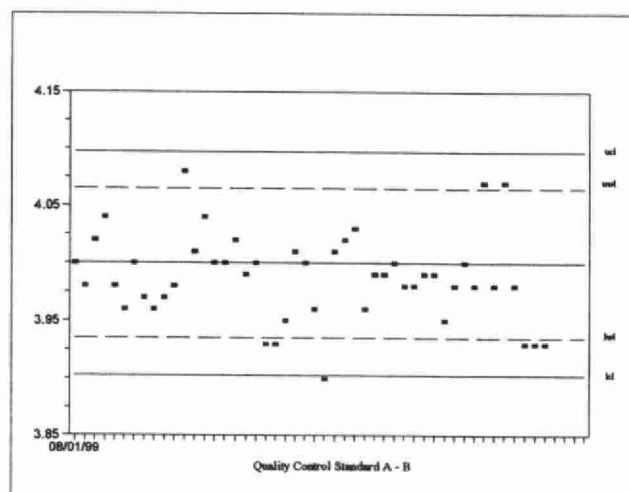
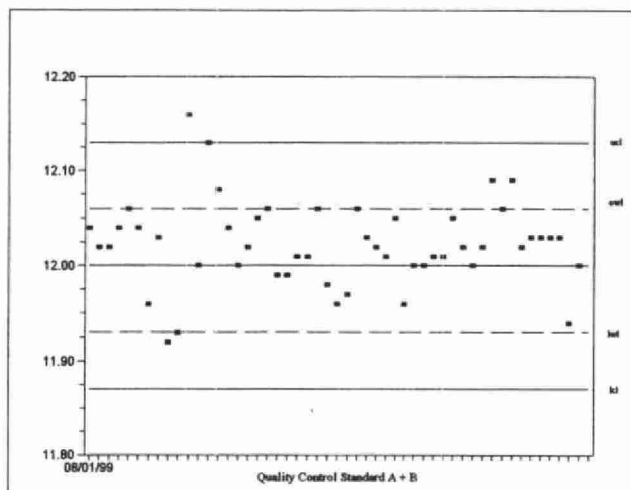
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
95	0.00 - 1.00	0.0623	36.7
20	1.00 - 2.00	0.2249	17.4
19	2.00 - 5.00	0.7701	25.4
6	5.00 - 10.00	0.9523	14.7
140	Overall	0.3613	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	51	0.004	0.0269
Digested Blank	51	0.008	0.0286

PHOSPHORUS, TOTAL (mg/L as P)
QUALITY CONTROL DATA FROM 08/01/99 TO 29/12/99
E3368



POTASSIUM

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	20/07/88
Method Reference No.	E3249	Reporting Unit	mg/L as K
LIMS Product Code	CAT3249	Supervisor	F. Tomassini
Sample Type/Matrix	Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	5 mL
Container	Plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 766.5 nm with an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level.

INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.005	Current T value: 0.025
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CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g., QCA
Drift	BL, reslope standard every 10 samples.

POTASSIUM (E3249)

QUALITY CONTROL DATA FROM 12/01/99 TO 22/12/99

Full Scale: to 1.0 mg/L as K

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	47	0.80	0.786	-0.0141	0.0091
B:	47	0.20	0.199	-0.0011	0.0035
C:	47	0.05	0.052	0.0024	0.0065
A+B:		1.00	0.985	-0.0152	0.0090
A-B:		0.60	0.587	-0.0129	0.0105
B+C:		0.25	0.251	0.0013	0.0085
B-C:		0.15	0.146	-0.0035	0.0062

s.d.(AB) S(between runs): 0.0069

Sw(within run): 0.0074

S/Sw: 0.93

s.d.(BC) S(between runs): 0.0046

Sw(within run): 0.0044

S/Sw: 1.05

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.966	-	1.034	for	A+B
0.574	-	0.626	for	A-B
0.222	-	0.278	for	B+C
0.129	-	0.171	for	B-C

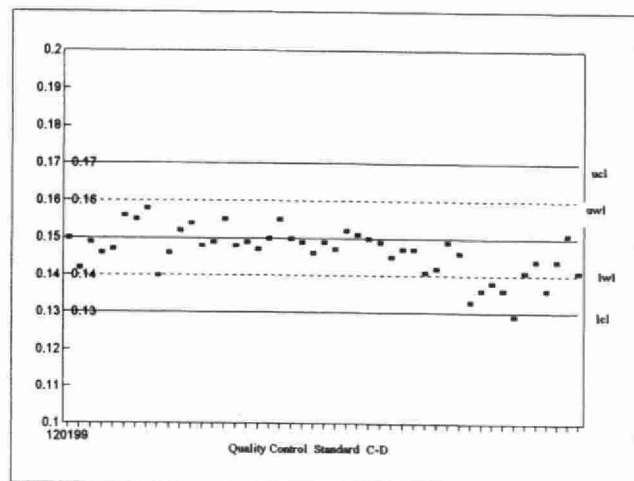
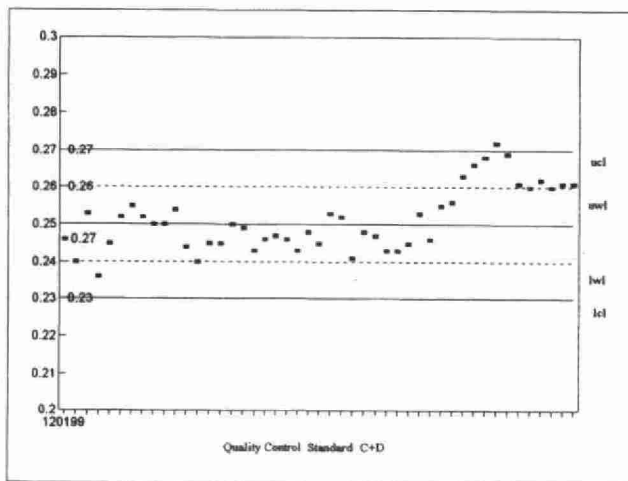
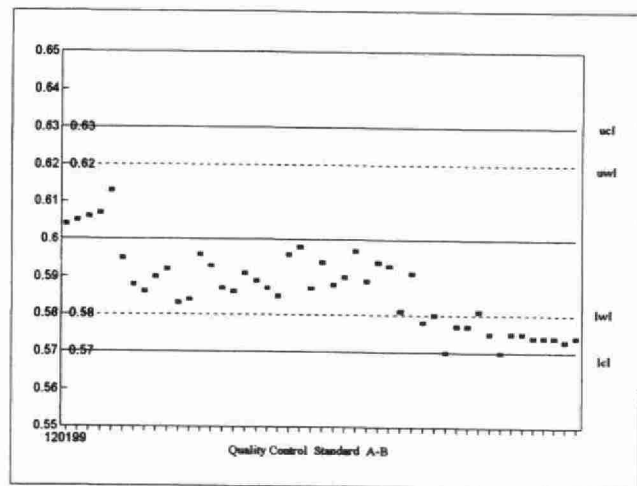
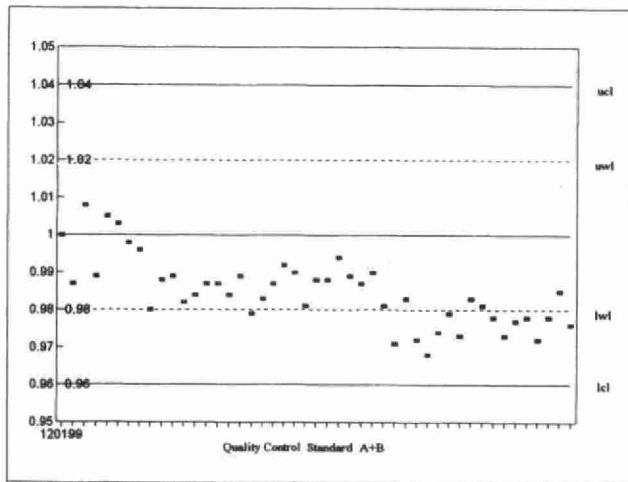
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
53	0.00 - 0.10	0.0070	13.9
23	0.11 - 0.20	0.0062	4.3
72	0.21 - 0.50	0.0065	1.9
26	0.51 - 1.00	0.0998	16.3
174	Overall	0.0390	14.5

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	47	0.0031	0.0081

POTASSIUM (mg/L as K)
 QUALITY CONTROL DATA FROM 12/01/99 TO 22/12/99
 E3249



SILICON, REACTIVE SILICATES

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/02/75
Method Reference No.	E3370	Reporting Unit	mg/L as Si
LIMS Product Code	DCSI3370	Supervisor	P.Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water Ground Water, Leachates, Precipitation, Surface Water		

SAMPLING:

Quantity Required	10 mL
Container	Plastic

ANALYTICAL PROCEDURE:

Reactive silicates are determined by formation of a reduced molybdo-silicate complex at pH 1.6, using ascorbic acid as the reducing agent, and oxalic acid to suppress phosphate interference.

Approximate absorbance: 0.7 at the full scale level.

Dissolved inorganic and dissolved organic carbon are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 660 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.02	Current T value: 0.10
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g., QCA
Drift	BL, standard and BL every 10 samples.

NOTES:

December 1998: The HP data capture/processing system was replaced by Labtronics.

SILICON, REACTIVE SILICATES (E3370)

QUALITY CONTROL DATA FROM 18/01/99 TO 17/12/99

Full Scale: to 10.0 mg/L as Si

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	57	8.00	8.011	0.011	0.0521
B:	57	2.00	2.011	0.011	0.0213
C:	57	0.50	0.488	-0.012	0.0111
A+B:		10.00	10.021	0.021	0.0640
A-B:		6.00	5.999	-0.001	0.0473
B+C:		2.50	2.499	-0.001	0.0293
B-C:		1.50	1.523	0.023	0.0226

s.d.(AB) S(between runs): 0.040

Sw(within run): 0.033

S/Sw: 1.4

s.d.(BC) S(between runs): 0.017

Sw(within run): 0.012

S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

9.66	-	10.34	for	A+B
5.75	-	6.25	for	A-B
2.37	-	2.63	for	B+C
1.40	-	1.60	for	B-C

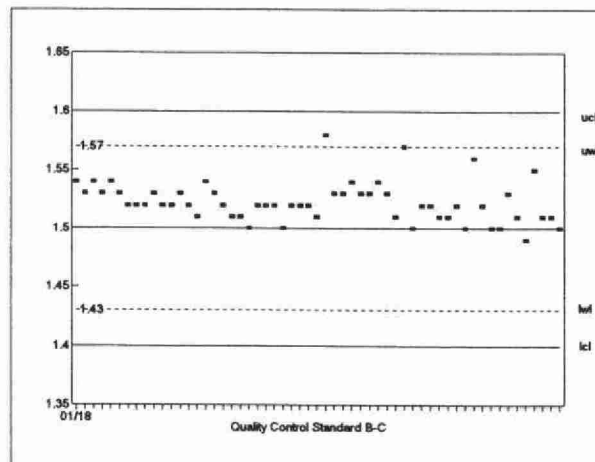
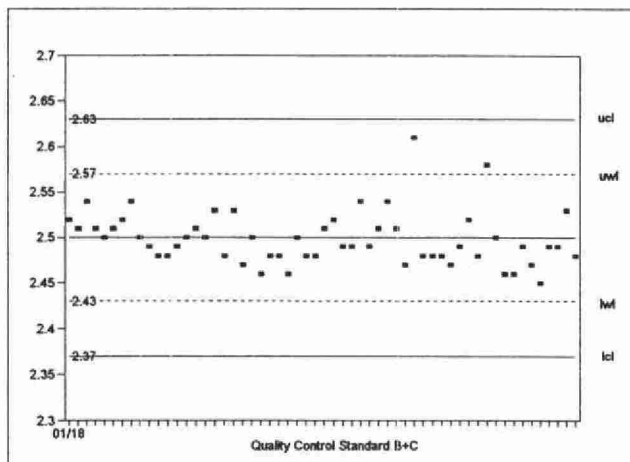
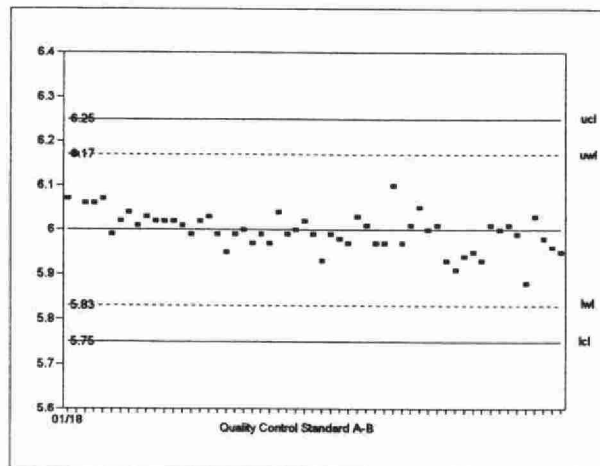
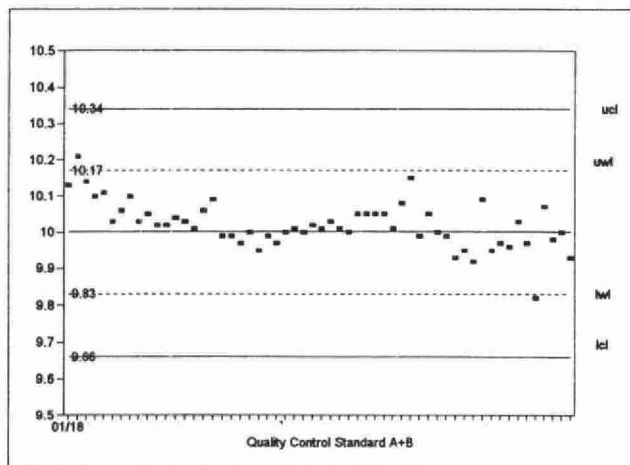
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
80	0.00 - 1.00	0.0078	1.4
53	1.01 - 2.00	0.0105	0.8
32	2.01 - 5.00	0.0219	0.7
6	5.01 -10.00	0.0176	0.3
171	Overall	0.0127	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	57	-0.0447	0.0720

SILICON, REACTIVE SILICATES (mg/L as Si)
QUALITY CONTROL DATA FROM 18/01/99 TO 17/12/99
E3370



SODIUM

IDENTIFICATION:

Laboratory Unit	Dorset	Method Introduced	20/07/88
Method Reference No.	E3249	Reporting Unit	mg/L as Na
LIMS Product Code	CAT3249	Supervisor	F. Tomassini
Sample Type/Matrix	Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	5 mL
Container	Plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 589.0 nm with an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level.

INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.005	Current T value: 0.025
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CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g., QCA
Drift	BL, reslope standard every 10 samples.

SODIUM (E3249)

QUALITY CONTROL DATA FROM 12/01/99 TO 22/12/99

Full Scale: to 4.0 mg/L as Na

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	47	3.2	3.197	-0.003	0.0195
B:	47	0.8	0.793	-0.007	0.0084
C:	47	0.2	0.199	-0.001	0.0075
A+B:		4.0	3.990	-0.010	0.0240
A-B:		2.4	2.404	0.004	0.0180
B+C:		1.0	0.992	-0.008	0.0136
B-C:		0.6	0.594	-0.006	0.0082

s.d.(AB) S(between runs): 0.015

Sw(within run): 0.013

S/Sw: 1.2

s.d.(BC) S(between runs): 0.005

Sw(within run): 0.006

S/Sw: 0.91

The calibration is accepted if the calibration control values obtained lie within the ranges:

3.929	-	4.071	for	A+B
2.347	-	2.453	for	A-B
0.968	-	1.032	for	B+C
0.576	-	0.624	for	B-C

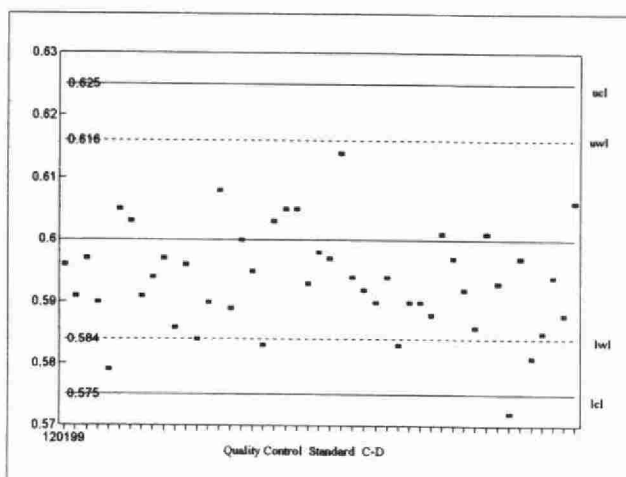
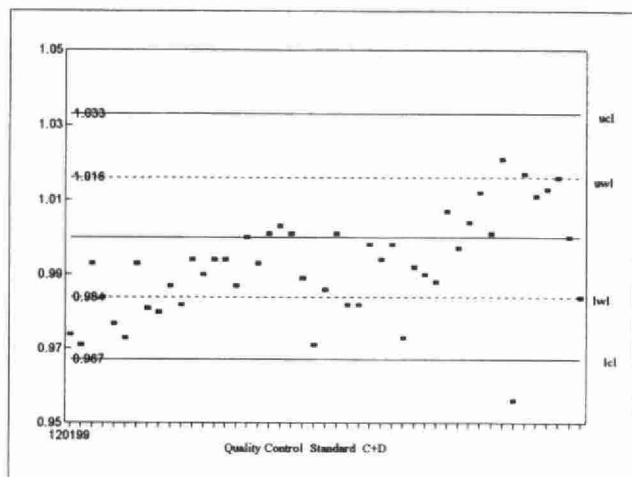
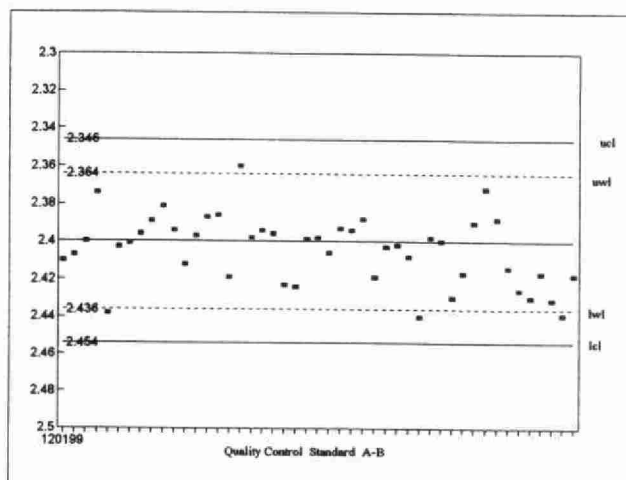
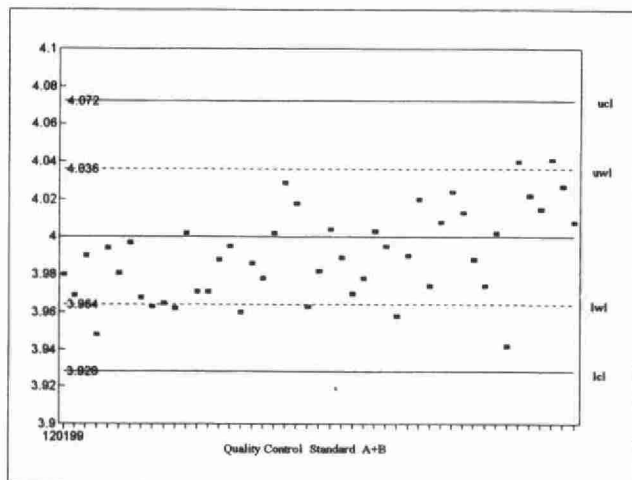
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
42	0.00 - 0.40	0.0049	5.1
77	0.41 - 0.80	0.0192	3.0
25	0.81 - 2.00	0.0210	1.8
34	2.01 - 4.00	0.0454	1.8
178	Overall	0.1564	2.7

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	47	-0.0031	0.010

SODIUM (mg/L as Na)
QUALITY CONTROL DATA FROM 12/01/99 TO 22/12/99
E3249



SOLIDS, DISSOLVED

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Before '61
Method Reference No.	E3188	Reporting Unit	mg/L
LIMS Product Code	TSD3188,DS3188,DIGN3188	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Surface Water, Drinking Water, Ground Water, Leachate		

SAMPLING:

Quantity Required	125 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Sample is filtered under moderate suction through a Whatman 934AH grade glass fibre filter. Generally 100 mL of filtrate (alternate 50 mL) is pipetted into a preweighed Teflon dish, dried at $103\pm 2^{\circ}\text{C}$, and stored in a desiccator for at least 24 hours. The dissolved solids content is calculated by subtracting the original dish mass from the dried residue + dish mass. Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

INSTRUMENTATION:

Balance (5 decimal places), drying oven, suction filtration apparatus, dishes (Teflon).
Microcomputer system with appropriate software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 2	Current T value: 10
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CALIBRATION:

Balance zero
Balance internal calibration is performed daily.

CONTROLS:

Calibration	2 S class weights, e.g. QCA (results in grams)
Drift	Balance is reset to zero after every 10 weighings by the microcomputer.
Recovery	2 standards, e.g. R1
Method Blank	100 mL distilled water.

SOLIDS, DISSOLVED (E3188)

(mg/L)

QUALITY CONTROL DATA FROM 11/01/99 TO 30/12/99

CALIBRATION CONTROL:

	n	Expected Mass (g)	Mean Mass Measured (g)	Mean Bias (g)	Standard Deviation (1)
A:	85	50.00	50.0006	0.0006	0.00006
B:	85	30.00	30.0002	0.0002	0.00005
A+B:		80.00	80.0008	0.0008	0.00010
A-B:		20.00	20.0004	0.0004	0.00005

s.d.(AB) S(between runs): 0.00005 Sw(within run): 0.00004 S/Sw: 1.5

The calibration is accepted if the calibration control values (mean mass measured) obtained lie within the ranges expressed in grams:

80.0005 - 80.0013 for A+B
20.0000 - 20.0006 for A-B

RECOVERIES:

Number of Data	Expected Concentration (mg/L)	Mean Concentration Measured (mg/L)	Standard Deviation (1)
85	2000.0	1984.82	8.8
85	500.0	487.09	6.4

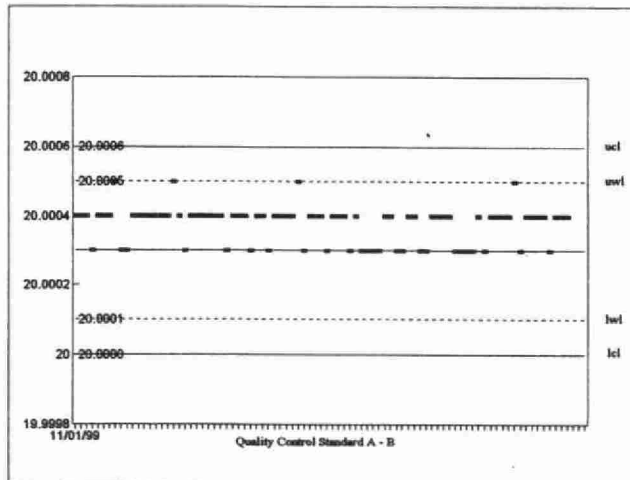
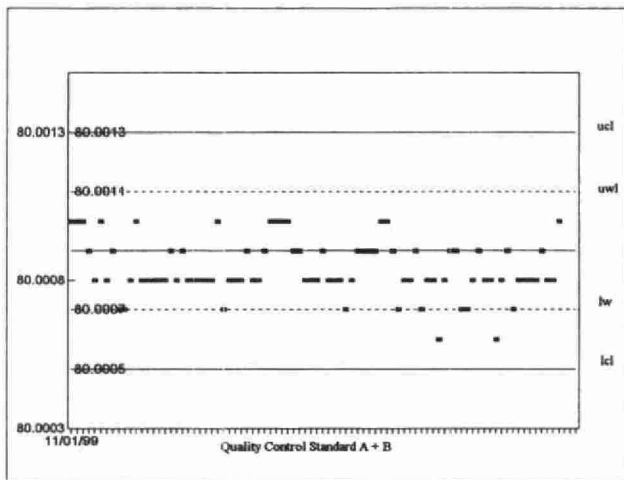
DUPLICATES:

n Data Pairs	Sample Concentration Span (mg/L)	Standard Deviation (2)	Coefficient of variation(%)
40	0 - 500	5.1984	1.7
138	501 - 1000	9.1097	1.3
32	1001 - 5000	37.7877	1.8
210	Overall	16.6514	

OTHER CHECKS:

	n	Data Mean (mg/L)	Standard Deviation (1)
Blank	82	-7.4957	6.3390

SOLIDS, DISSOLVED (mg/L)
QUALITY CONTROL DATA FROM 11/01/99 TO 30/12/99
E3188



SOLIDS, SUSPENDED

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Before '81
Method Reference No.	E3188	Reporting Unit	mg/L
LIMS Product Code	TSD3188, SS3188	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Surface Water, Drinking Water, Ground Water, Leachate		

SAMPLING:

Quantity Required	5-500 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

An appropriately shaken sample volume (5 to 500 mL) is pipetted or quickly poured into a graduated cylinder, and the volume is measured. The aliquot is then filtered under moderate suction through a preweighed Whatman 934AH glass fibre filter. The graduated cylinder and then the filter are washed with a total of 50 mL distilled water. The filter is dried at 103-105°C, and suspended solids content is calculated by subtracting the original filter mass from the dried filter mass. Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

INSTRUMENTATION:

Balance (5-decimal places), drying oven, suction filtration apparatus.
Microcomputer system with appropriate software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.5	Current T value: 2.5
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CONTROLS:

Calibration	2 S class weights, e.g. QCA (results in grams)
Drift	Balance is reset to zero after every 10 weighings by the microcomputer.
Recovery	2 standards, e.g. R1
Method Blank	Filter washed with 500 mL distilled water

NOTES:

A standard correction factor (-0.00022g) was applied to all filters to account for weight loss during filtering.

SOLIDS, SUSPENDED (E3188)
(mg/L)

QUALITY CONTROL DATA FROM 07/01/99 TO 30/12/99

CALIBRATION CONTROL: (QC data from SS3188)

	n	Expected Mass (g)	Mean Mass Measured (g)	Mean Bias (g)	Standard Deviation (1)
C:	259	0.50	0.4987	-0.0013	0.00001
D:	259	0.05	0.0500	0.0000	0.00002
C+D:		0.55	0.5487	-0.0013	0.00002
C-D:		0.45	0.4487	-0.0013	0.00002

s.d.(CD) S(between runs): 0.00002 Sw(within run): 0.00002 S/Sw: 1.00

The calibration is accepted if the calibration control values (mean mass measured) obtained within the ranges expressed in grams:

0.54984 - 0.55000 for C+D
0.44979 - 0.44991 for C-D

RECOVERIES:

Number of Data	Expected Concentration (mg/L)	Mean Concentration Measured (mg/L)	Standard Deviation (1)
256	200.0	194.10	2.5786
255	50.0	49.49	1.3101

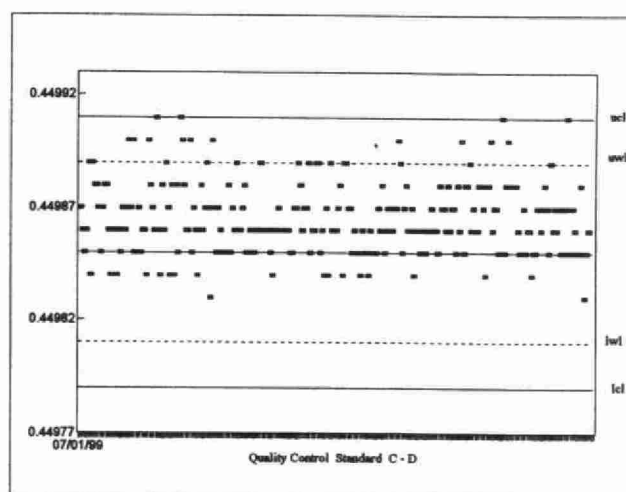
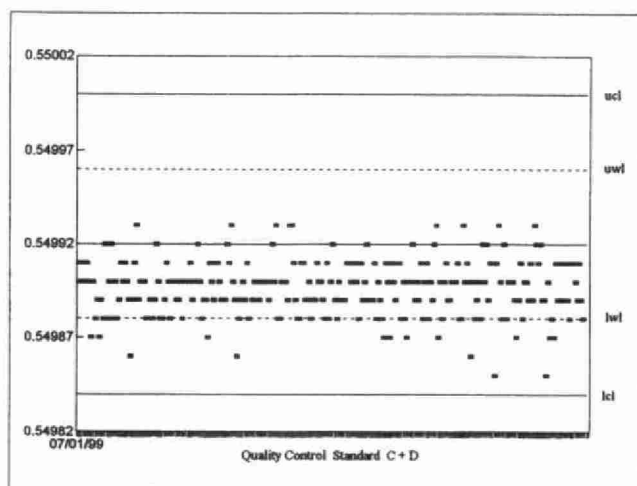
DUPLICATES:

n Data Pairs	Sample Concentration Span (mg/L)	Standard Deviation (2)	Coefficient of variation(%)
199	0.0 - 5.0	0.3167	13.2
133	5.1 - 10	0.5745	8.2
121	10.1 - 25	1.2625	8.6
132	25.1 - 100	3.7862	7.6
81	100.1 - 500	9.4768	4.8
10	500.1 - 1000	21.3915	2.6
17	1000.1 - 10000	49.5555	0.7
693	Overall	8.9690	

OTHER CHECKS:

	n	Data Mean (mg/L)	Standard Deviation (1)
Blank	257	0.1156	0.2275

SOLIDS, SUSPENDED (mg/L)
QUALITY CONTROL DATA FROM 07/01/99 TO 30/12/99
E3188



SOLIDS, SUSPENDED IGNITED
(Particulate Ash and Particulate Loss On Ignition)

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Before '61
Method Reference No.	E3188	Reporting Unit	mg/L
LIMS Product Code	SIGN3188	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water		

SAMPLING:

Quantity Required	5-500 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

The procedure for particulate solids (SS3188) is followed and the dried residue is ignited at $600 \pm 50^{\circ}\text{C}$ for one hour in a muffle furnace. The dish is transferred to a desiccator to cool. The particulate ash (fixed solids) is the difference between the final ignited mass plus filter and the original tare weight of the filter, divided by the original sample volume (mL) used for SS3188. The particulate loss on ignition (estimate of volatile suspended solids) is the difference between the final ignited mass plus filter and the residue (suspended solids) plus filter, divided by the original sample volume (mL). Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

INSTRUMENTATION:

Balance (5 decimal places), muffle furnace, filters, Petri dishes
 Microcomputer system with appropriate software

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.5	Current T value: 2.5
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CONTROLS:

Calibration	2 S class weights, e.g. QCA (results in grams)
Drift	Balance is reset to zero after every 10 weighings by the microcomputer.

NOTE:

There is insufficient quality control data for year 1999.

SOLIDS, TOTAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Before '81
Method Reference No.	E3188	Reporting Unit	mg/L or mg/Kg
LIMS Product Code	TS3188	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Surface Water, Drinking Water, Ground Water, Leachate		

SAMPLING:

Quantity Required	125 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Generally, 100 mL aliquot of sample (alternate 50 mL) is pipetted into a preweighed Teflon dish, dried at 103-105°C, and stored in a desiccator for at least 24 hours. The total residue or solids content is calculated by subtracting the original dish mass from the dried dish mass. Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

INSTRUMENTATION:

Balance (5 decimal places), drying oven, dishes (Teflon).

Microcomputer system with appropriate software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 2.0	Current T value: 10
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CALIBRATION:

Balance zero

Balance internal calibration performed daily.

CONTROLS:

Calibration	2 S class weights, e.g. QCA (results in grams)
Drift	Balance is reset to zero after every 10 weighings by the microcomputer.
Recovery	2 standards, e.g. R1

SOLIDS, TOTAL (E3188)
(mg/L or mg/Kg)

QUALITY CONTROL DATA FROM 05/01/99 TO 23/12/99

CALIBRATION CONTROL: (QC data from TS3188)

	n	Expected Mass (g)	Mean Mass Measured (g)	Mean Bias (g)	Standard Deviation (1)
A:	15	50.00	50.0006	0.0006	0.00005
B:	15	30.00	30.0002	0.0002	0.00005
A+B:		80.00	80.0008	0.0008	0.00008
A-B:		20.00	20.0003	0.0003	0.00006

s.d.(AB) S(between runs): 0.00006 Sw(within run): 0.00004 S/Sw: 1.3

The calibration is accepted if the calibration control values (mean mass measured) obtained lie within the ranges expressed in grams:

80.0005 - 80.0013 for A+B
20.0000 - 20.0006 for A-B

RECOVERIES:

Number of Data	Expected Concentration (mg/L)	Mean Concentration Measured (mg/L)	Standard Deviation (1)
15	20000.0	20032.09	79.85
15	2000.0	1985.21	9.8

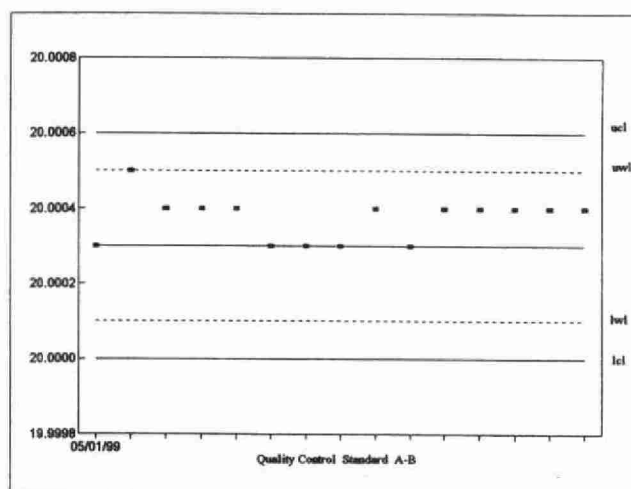
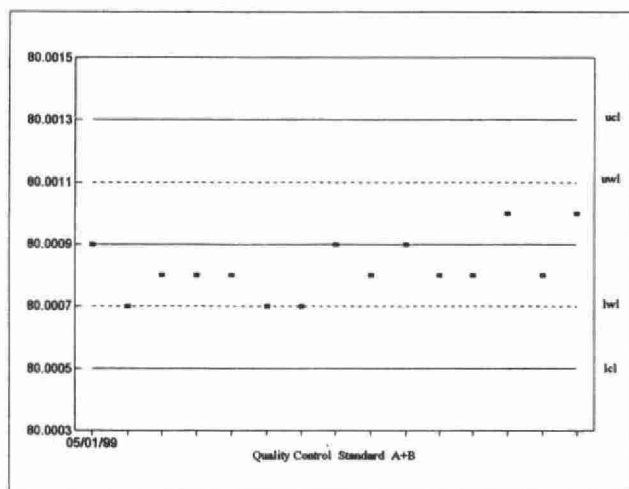
DUPLICATES:

n Data Pairs	Sample Concentration Span (mg/L)	Standard Deviation (2)	Coefficient of variation(%)
11	0 - 6000	29.7602	2.3
12	6001 - 25000	118.2857	0.7
8	25001 - 50000	124.9717	0.4
31	Overall	98.7966	

OTHER CHECKS:

	n	Data Mean (mg/L)	Standard Deviation (1)
Blank	15	-8.7067	4.0393

SOLIDS, TOTAL (mg/L)
QUALITY CONTROL DATA FROM 05/01/99 TO 23/12/99
E3188



SOLIDS, TOTAL IGNITED
(Ash and Loss On Ignition)

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Before '61
Method Reference No.	E3188	Reporting Unit	mg/L
LIMS Product Code	TIGN3188	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water		

SAMPLING:

Quantity Required	5-500 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

The procedure for total solids (TS3188) is followed and the dried residue is ignited at $600 \pm 50^{\circ}\text{C}$ for one hour in a muffle furnace. The dish is transferred to a desiccator to cool. The ash (fixed solids) is the difference between the final ignited mass plus filter and the original tare weight of the filter, divided by the original sample volume (mL) used for TS3188. The loss on ignition (estimate of volatile total solids) is the difference between the final ignited mass plus filter and the residue (total solids) plus filter, divided by the original sample volume (mL). Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

INSTRUMENTATION:

Balance (5 decimal places), muffle furnace, filters, Petri dishes.
Microcomputer system with appropriate software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 2.0	Current T value: 10
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CONTROLS:

Calibration	2 S class weights, e.g. QCA (results in grams)
Drift	Balance is reset to zero after every 10 weighings by the microcomputer.

SOLIDS, TOTAL IGNITED (E3188)
(Ash and Loss On Ignition)
(mg/L)

QUALITY CONTROL DATA FROM 11/01/99 TO 22/12/99

CALIBRATION CONTROL:

	n	Expected Mass (g)	Mean Mass Measured (g)	Mean Bias (g)	Standard Deviation (1)
A:	29	50.00	50.0006	0.0006	0.00007
B:	29	30.00	30.0002	0.0002	0.00006
A+B:		80.00	80.0008	0.0008	0.00011
A-B:		20.00	20.0004	0.0004	0.00005

s.d.(AB) S(between runs): 0.00006 Sw(within run): 0.00003 S/Sw: 1.8

The calibration is accepted if the calibration control values (mean mass measured) obtained lie within the ranges expressed in grams:

80.0005 - 80.0013 for A+B
20.0000 - 20.0006 for A-B

SOLIDS, TOTAL IGNITED (DRY)

RECOVERIES:

Number of Data	Expected Concentration (mg/L)	Mean Concentration Measured (mg/L)	Standard Deviation (1)
28	20000.0	19868.03	154.3
29	2000.0	1972.48	12.1

DUPLICATES:

n Data Pairs	Sample Concentration Span (mg/L)	Standard Deviation (2)	Coefficient of variation(%)
0	0 - 5000	N.A.	N.A.
3	5001 - 15000	118.6	0.93
61	15001 - 40000	284.2	1.11
64	Overall	277.9	

SOLIDS, TOTAL IGNITED cont'd
(Ash and Loss On Ignition)
(mg/L)

SOLIDS, TOTAL IGNITED (ASH)

DUPLICATES:

n Data Pairs	Sample Concentration Span (mg/L)	Standard Deviation (2)	Coefficient of variation(%)
0	0 - 3000	N.A.	N.A.
27	3001 - 10000	109	1.3
20	10001 - 15000	158	1.3
26	15001 - 25000	293	1.3
73	Overall	204	

OTHER CHECKS:

Ashed	n	Data Mean (mg/L)	Standard Deviation (1)
Blank	29	-17.736	5.1366

SOLIDS, TOTAL IGNITED (LOSS ON IGNITION)

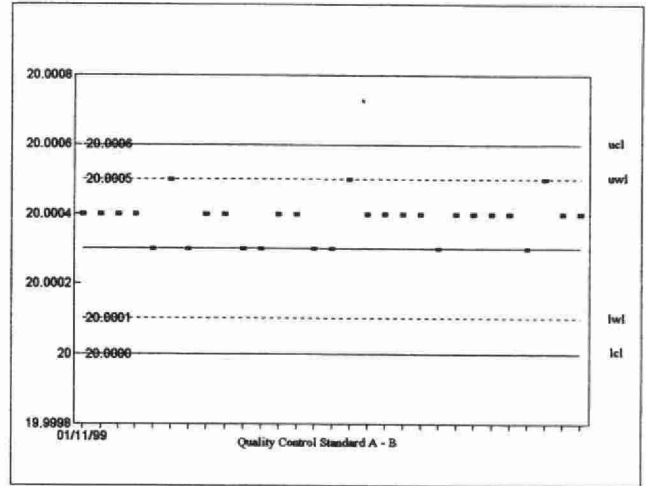
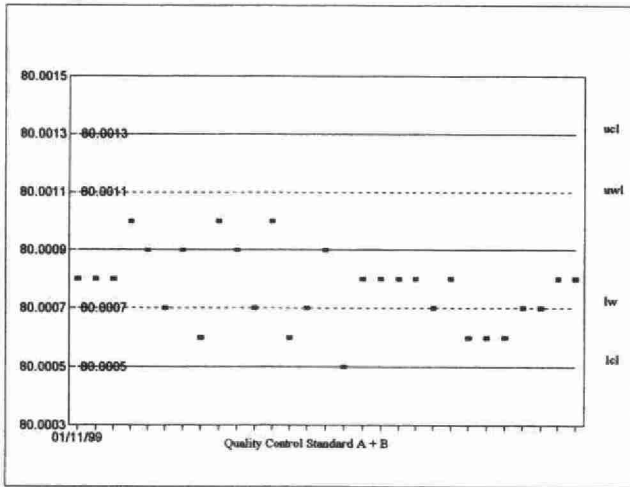
DUPLICATES:

n Data Pairs	Sample Concentration Span (mg/L)	Standard Deviation (2)	Coefficient of variation(%)
0	0 - 5000	NA	NA
36	5001 - 15000	263	2.3
26	15001 - 25000	315	1.5
10	25001 - 50000	505	1.7
72	Overall	325	

OTHER CHECKS:

LOI	n	Data Mean (mg/L)	Standard Deviation (1)
Blank	29	-0.3428	4.8551

SOLIDS, TOTAL IGNITED (mg/L)
ASH AND LOSS ON IGNITION
QUALITY CONTROL DATA FROM 11/01/99 TO 22/12/99
E3188



SULPHATE

IDENTIFICATION:

Laboratory Unit	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3004	Units	$\mu\text{g}/\text{m}^3$ as SO_4
LIMS Product Code	ANION3004	Supervisor	P. Wilson
Sample Type/Matrix	Air, HiVol Glass Fibre, Quartz and Polyflon, Other Filters and Puff		

SAMPLING:

Quantity Required	3/4" or 1.9cm strip from 8"x10" filter
Container	50 mL polypropylene tube

SAMPLING PREPARATION:

A 3/4" strip is cut in pieces and deposited into a 50 mL polypropylene tube. 50 mL of Pure-Water is added to the tube. The tube is placed on a horizontal shaker for approximately 1 hour. The supernatant is then filtered into a 15 mL plastic tube and analysed.

ANALYTICAL PROCEDURE:

Sulphate separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of sodium bicarbonate and sodium carbonate and a conductivity detector. The concentration of sulphate (mg/L) is determined by the comparison of the analyte peak area count to that of a series of standards. The analyte result is corrected for the filter blank before the final calculation is made. The result is reported as $\mu\text{g}/\text{m}^3$ as SO_4 . Chloride and nitrate are determined simultaneously.

INSTRUMENTATION:

Horizontal Shaker, ion chromatographic system plus a PC with ChromPerfect software and DT2804 card for automated sample injection, timing, and data processing.

REPORTING:

Maximum Significant Figures: 2	Current W value: $0.1 \mu\text{g}/\text{m}^3$	Current T value: $0.5 \mu\text{g}/\text{m}^3$
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CALIBRATION:

6 standards

CONTROLS:

Calibration	MB, IS(n), CS1, and CS2
Drift	Duplicate plus 2 standards approximately every 20 samples

SULPHATE cont'd

NOTES:

To convert unit from mg/L to $\mu\text{g}/\text{m}^3$, the final concentration of SO_4 in mg/L is multiplied by the following formula:

$$\text{Result (mg/L)} \times 50\text{mL} \times (63/6.75) / \text{air volume} = \mu\text{g}/\text{m}^3$$

Where: 63 is the area of the filter exposed and 6.75 is the sample aliquot area in square inch.

SULFATE (E3004)

QUALITY CONTROL DATA FROM 1997 TO 1999

Analytical Range: to 28.61 $\mu\text{g}/\text{m}^3$

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
73	0.00 - 2.86	0.066	3.9
64	2.89 - 7.15	0.123	2.9
43	7.18 - 14.31	0.114	1.1
13	14.33 - 24.90	0.114	0.9
193	Overall	0.105	

SULPHATE

IDENTIFICATION:

Laboratory Unit	Water Chemistry	Method Introduced	01/01/86
Method Reference No.	E3013	Units	µg/g as SO ₄
LIMS Product Code	ANION3013, SUL3013	Supervisor	P. Wilson
Sample Type/Matrix	Soil and Sediment		

SAMPLING:

Quantity Required	20 g
Container	glass or plastic

SAMPLING PREPARATION:

A 3.0 g sample air dried, sieved soil or air dried sieved and ground sediment is placed in a 50 mL centrifuge tube and shaken with 30 mL Pure-DW for 1 hour on a shaker. Samples are centrifuged, membrane filtered and analyzed for chloride and sulphate by ion chromatography.

ANALYTICAL PROCEDURE:

Sulphate separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of sodium bicarbonate and sodium carbonate and a conductivity detector. The concentration of sulphate (mg/L) is determined by the comparison of the analyte peak area count to that of a series of standards. The result is reported as µg/g as SO₄.

Chloride is determined simultaneously.

INSTRUMENTATION:

Horizontal Shaker, ion chromatographic system plus a PC with ChromPerfect Sprit software and DT2804 card for automated sample injection, timing, and data processing.

REPORTING:

Maximum Significant Figures: 2	Current W value: 0.5 µg/g	Current T value: 2.5 µg/g
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CALIBRATION:

8 standards

CONTROLS:

Calibration	IS(n) - certified reference check solution, CS1, CS2, - check standards, in house matrix check standards MC(n)
Drift	Duplicate plus 2 check standards approximately every 20 samples
Contamination	Method Blank (MB)
Interference	SP1 and SP2 (spike solutions) for positive and negative interferences

SULFATE (E3013)

QUALITY CONTROL DATA FROM 1998 TO 2000

Analytical Range: to 1000 µg/g

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
35	0 - 200	2.5731	7.1
3	201 - 500	4.5055	1.6
4	501 - 1000	9.4112	1.1
42	Overall	3.9246	

SULPHATE

IDENTIFICATION:

Laboratory	Dorset	Method Introduced	01/04/78
Method Reference No.	E3147	Reporting Unit	mg/L as SO ₄
LIMS Product Code	ANION3147	Supervisor	F. Tomassini
Sample Type/Matrix	Precipitation, Leachate, Surface Water		

SAMPLING:

Quantity Required	15 mL
Container	Glass or Plastic

ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO₄ is determined by the comparison of the sample peak heights to a series of standards.

Chloride is determined simultaneously.

INSTRUMENTATION:

Modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing, and partial data processing.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.05	Current T value: 0.25
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 2 standards, e.g., QCA
Drift	1 standard every 10 samples.

SULPHATE (E3147)

QUALITY CONTROL DATA FROM 14/01/99 TO 15/12/99

Full Scale: to 10.0 mg/L as SO₄

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	25	8.0	8.078	0.078	0.0897
B:	25	2.0	1.978	-0.022	0.0596
A+B:		10.0	10.056	0.056	0.1329
A-B:		6.0	6.099	0.099	0.0744

s.d.(AB)

S(between runs): 0.076

Sw(within run): 0.053

S/Sw: 1.4

The calibration is accepted if the calibration control values obtained lie within the ranges:

9.70 - 10.30 for A+B

5.77 - 6.23 for A-B

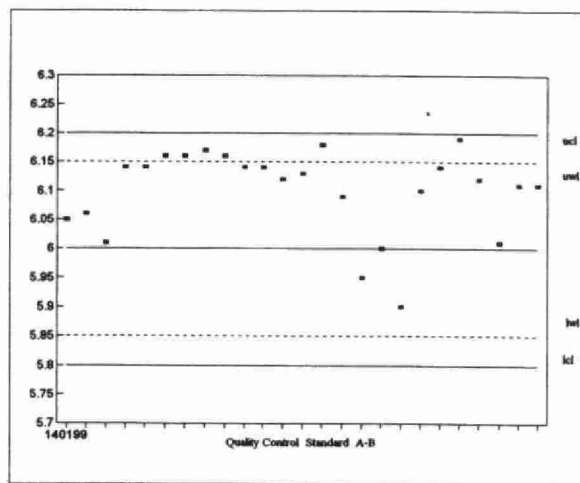
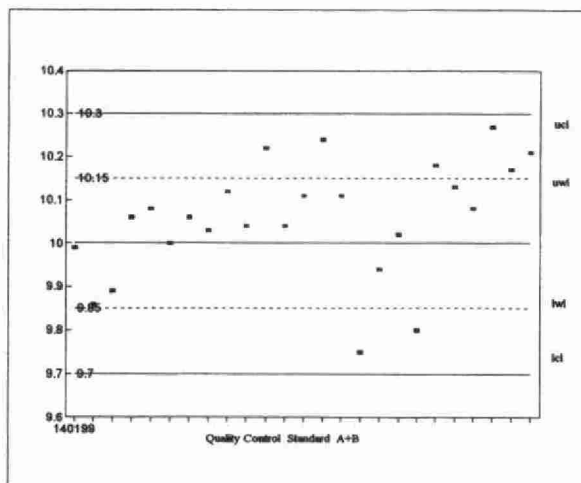
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
20	0.00 - 2.00	0.0497	4.0
29	2.01 - 5.00	0.0946	2.9
33	5.01 - 10.0	0.2742	4.1
82	Overall	0.1844	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	25	0.0208	0.0320

SULPHATE (mg/L as SO₄)
QUALITY CONTROL DATA FROM 14/01/99 TO 15/12/99
E3147



SULPHATE

IDENTIFICATION:

Laboratory Unit	Water Chemistry	Method Introduced	01/04/78
Method Reference No	E3148	Reporting Unit	µg/Filter as SO ₄
LIMS Product Code	LOV3148, ANLOV3148, TEF3148, NYL3148, SDIO3148, ANION3148	Supervisor	P. Wilson
Sample Type/Matrix	Air; Sequential and LoVol filters		

SAMPLING:

Quantity Required	1 filter for W40, Teflon or Nylon. 1 set of 2 W41 filters
Container	50 mL polypropylene tube
Other	For W41 filters, filters are impregnated with potassium carbonate / glycerol solution

SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of Pure-DW (W40) or 25.0 mL of Pure-DW (Teflon) or 25.0 mL of 0.03 N NaOH (Nylon) in polypropylene tubes with ultrasonic treatment followed by a 24 hour rest period. For W41 filters, filters are extracted with 50.0 mL of 0.05% H₂O₂ in polypropylene tubes with one hour of mechanical shaking, followed by ultrasonic treatment to enhance extraction, then a 24 hour rest period. SO₂ is converted to SO₄ in the process.

ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO₄ is determined by the comparison of the sample peak heights to a series of standards. Results are converted to µg/filter as SO₄ for W40, Teflon and Nylon filters. As for W41 filters, results are converted to µg/filter as SO₄.

Chloride and nitrogen-nitrate are determined simultaneously.

INSTRUMENTATION:

Mechanical shaker, Ultrasonic bath; modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing and partial data processing.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.02 mg/L	Current T value: 0.1 mg/L
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CALIBRATION:

BL plus 9 standards

SULPHATE cont'd

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
Drift	1 standard every 10 samples

NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received. To convert unit from mg/L to $\mu\text{g}/\text{Filter}$, multiply the concentration of SO_4 in mg/L by 50 for W40 filters or 25 for Teflon or Nylon filters or 33.3 for W41 filters.

SULPHATE (E3148)

QUALITY CONTROL DATA FROM 26/01/99 TO 06/12/99

Full Scale: to 10.0 mg/L as SO₄

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	18	8.0	7.946	-0.054	0.1043
B:	18	2.0	2.006	0.006	0.0829
A+B:		10.0	9.951	-0.049	0.1477
A-B:		6.0	5.940	-0.060	0.1170

s.d.(AB)

S(between runs): 0.094

Sw(within run):0.083

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

9.68 - 10.32 for A+B
5.76 - 6.24 for A-B

DUPLICATES:

For W40 filters:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
8	0 - 2.00	0.0418	2.4
14	2.01 - 5.00	0.0588	1.6
3	5.01 - 10.00	0.0676	1.0
25	Overall	0.0552	

For Teflon filters:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
17	0 - 1.00	0.0229	4.2
5	1.01 - 2.00	0.0422	2.2
11	2.01 - 5.00	0.0453	1.4
2	5.01 - 10.00	0.1012	1.0
35	Overall	0.0417	

SULPHATE cont'd (E3148)

QUALITY CONTROL DATA FROM 26/01/99 TO 06/12/99

Full Scale: to 10.0 mg/L as SO₄

For Nylon filters:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
30	0 - 1.00	0.0193	9.9
4	1.01 - 2.00	0.0444	2.8
8	2.01 - 5.00	0.0810	3.1
2	5.01 - 10.00	0.0901	1.6
44	Overall	4.4975	

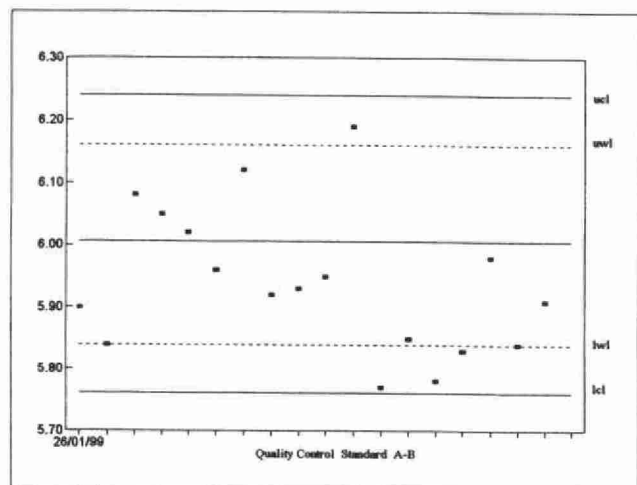
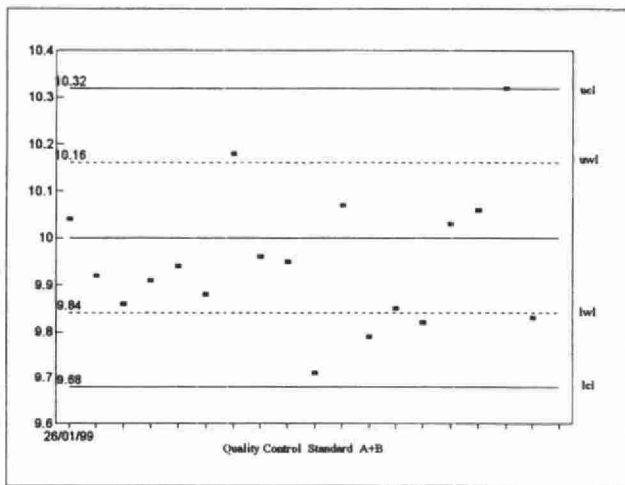
For W41 filters:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
16	0 - 1.00	0.0519	18.9
9	1.01 - 2.00	0.0432	4.0
13	2.01 - 5.00	0.1355	4.3
10	5.01 - 10.00	0.1111	1.4
48	Overall	0.0938	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	18	0.0433	0.0859

SULPHATE (mg/L as SO₄)
QUALITY CONTROL DATA FROM 26/01/99 TO 06/12/99
E3148



SULPHATE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/82
Method Reference No.	E3172	Reporting Unit	mg/L as SO ₄
LIMS Product Code	SULP3172	Supervisor	P.Wilson
Sample Type/Matrix	Drinking Water, Surface Water, Ground Water, Leachates, Effluent, Industrial Waste, Raw Sewage		

SAMPLING:

Quantity Required	50 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the samples by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. The concentration of sulphate in mg/L as SO₄ is determined by comparison of the sample scan to a series of standard scans.

INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system plus control module (in-house design) for automated sample introduction and timing.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.5	Current T value: 2.5
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CALIBRATION:

BL plus 10 standards

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
Drift	1 standard every 10 samples

SULPHATE (E3172)

QUALITY CONTROL DATA FROM 08/01/99 TO 23/12/99

Full Scale: to 100.0 mg/L as SO₄

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	34	80.0	80.7	0.7	0.7257
B:	34	20.0	20.2	0.2	0.6409
A+B		100.0	100.9	0.9	1.1804
A-B		60.0	60.5	0.5	0.6940

s.d.(AB) S(between runs): 0.68 Sw(within run): 0.49 S/Sw: 1.39

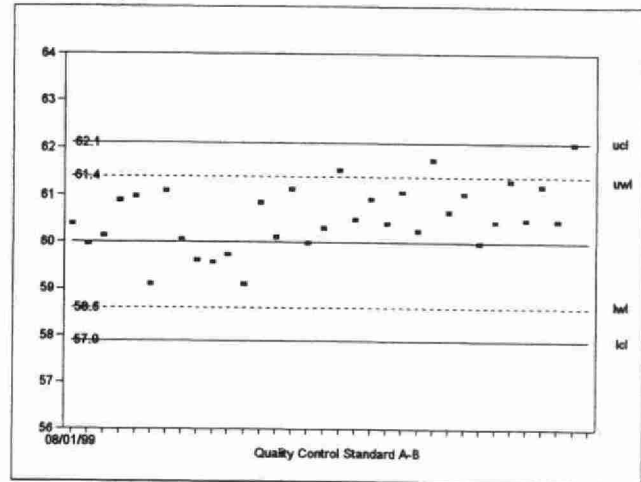
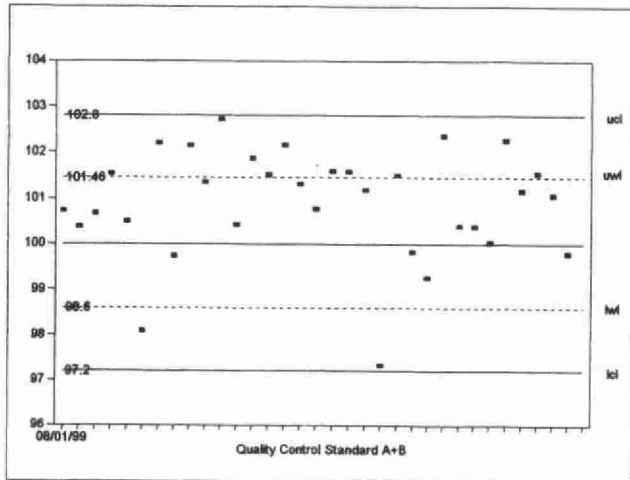
The calibration is accepted if the calibration control values obtained lie within the ranges:

97.2 - 102.8 for A+B
57.9 - 62.1 for A-B

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
21	0.0 - 10.0	0.3537	7.5
20	10.1 - 20.0	0.3568	2.3
35	20.1 - 50.0	0.6504	2.3
15	50.1 - 100.0	0.8970	1.3
91	Overall	0.5935	

SULPHATE (mg/L as SO₄)
QUALITY CONTROL FROM 08/01/99 TO 23/12/99
E3172



SULPHATE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No	E3372	Reporting Unit	mg/L as SO ₄
LIMS Product Code	ANION3372	Supervisor	P.Wilson
Sample Type/Matrix	Precipitation		

SAMPLING:

Quantity Required	15 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO₄ is determined by the comparison of the sample peak heights to a series of standards.

Chloride and nitrogen-nitrate are determined simultaneously.

INSTRUMENTATION:

Modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing, and partial data processing.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.05	Current T value: 0.25
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
Drift	1 standard every 10 samples

SULPHATE (E3372)

QUALITY CONTROL DATA FROM 19/01/99 TO 23/12/99

Full Scale: to 5.0 mg/L as SO₄

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	20	4.00	3.993	-0.007	0.0553
B:	20	1.00	0.962	-0.038	0.0799
A+B:		5.00	4.955	-0.045	0.0871
A-B:		3.00	3.031	0.032	0.1063

s.d.(AB) S(between runs): 0.069

Sw(within run): 0.075

S/Sw: 0.91

The calibration is accepted if the calibration control values obtained lie within the ranges:

4.79 - 5.21 for A+B
2.84 - 3.16 for A-B

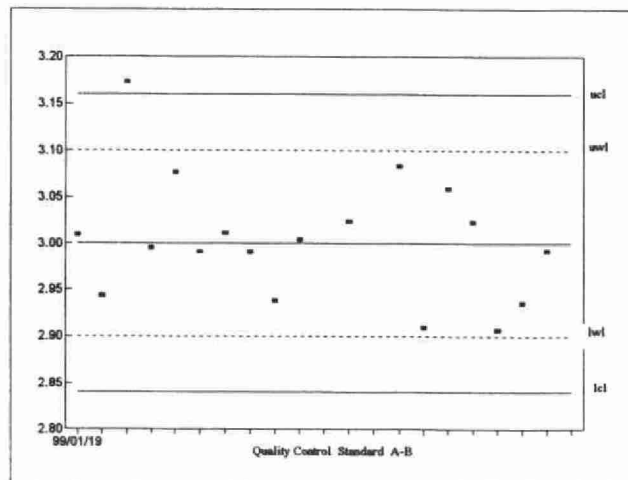
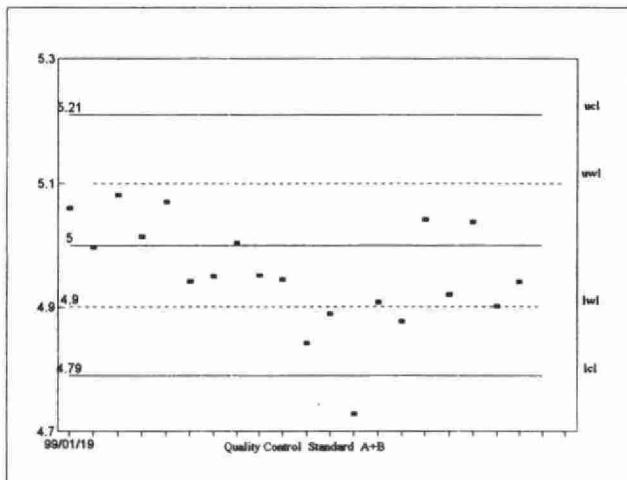
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
5	0 - 1.00	0.0394	9.1
15	1.01 - 2.50	0.0255	1.5
19	2.50 - 5.00	0.0435	1.3
39	Overall	0.0370	1.6

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	20	0.045	0.0104

SULPHATE (mg/L as SO₄)
QUALITY CONTROL DATA FROM 19/01/99 TO 23/12/99
E3372



SULPHIDE, TOTAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/01/88
Method Reference No.	E3100	Reporting Unit	µg/L as S ²⁻
LIMS Product Code	H2S3100	Supervisor	P. Wilson
Sample Type/Matrix	Industrial Waste, Process Water, Raw Sewage, Drinking Water, Ground Water, Landfill Leachate, Surface Water		

SAMPLING:

Quantity Required:	50 mL
Container:	glass or plastic

ANALYTICAL PROCEDURE:

This method is used to determine **Total Sulphide (S²⁻)** which includes hydrogen sulphide H₂S and HS⁻ (both of which will have been precipitated as ZnS during sample preservation), and any acid soluble metal sulphides. The addition of an absorbing solution to the sample dissolves the precipitates. Addition of acid to the sample releases the sulphides as hydrogen sulphide which is then stripped from the sample by nitrogen gas in a continuous flow system. The hydrogen sulphide is then trapped in an alkaline absorbing solution of sodium carboxymethyl cellulose and cadmium sulphate and reacted with N,N-dimethyl-p-phenylenediamine dihydrochloride and ferric chloride to form methylene blue. The intensity of the methylene blue is measured at 660 nm, and compared to standards treated in the same manner.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 1.5 mm light path at 660 nm.

The peak heights of the samples and the standards are read directly from the chart recorder.

REPORTING:

Maximum Significant Figures: 3	Current W value: 2.0	Current T value: 10.0
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CALIBRATION:

Preserved blank plus 4 preserved standards

CONTROLS:

Calibration:	LTBL plus 3 standards, e.g. QCA
Drift:	Standard after every 10 samples and blank after every sample

Notes: Data summary for '99 not available due to insufficient data.

TURBIDITY

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Before'74
Method Reference No.	E3311	Reporting Unit	FTU
LIMS Product Code	TURB3311	Supervisor	P. Wilson
Sample Type/Matrix	Surface Water, Ground Water, Effluent, Drinking Water, Industrial Waste, Process Water, Leachate		

SAMPLING:

Quantity Required:	50 mL
Container:	Glass or plastic

ANALYTICAL PROCEDURE:

The instrument is standardized with sealed standards which are prepared commercially and rated in Formazin Turbidity Units. Samples are placed in the turbidimeter, and results in FTU are read directly from the digital output. Turbidity measurement are based on light scattering at 90 plus or minus 30 degrees of rotation. The instrument compensates for sample colour.

INSTRUMENTATION:

-Hach Ratio/XR Model Turbidimeter modified to accept control signals from robot controller, electronic interphase, Zymark ZYMATE 11 Laboratory Robot System, IBM PC computer.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.05	Current T value: 0.25
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CALIBRATION:

BL plus formazin standards (once every four months)

CONTROLS:

Calibration:	5 standards, e.g. QCA
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TURBIDITY (E3311)

QUALITY CONTROL DATA FROM 20/01/99 TO 31/12/99

Full Scale: to 2000 FTU

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Standard Deviation (1)
A:	159	2.0	1.57	0.0223
B:	158	20.0	15.41	0.1490
C:	159	200.0	147.93	0.8207
D:	143	2000.0	1352.85	47.8134

On any given day the calibration is accepted if the values obtained lie within the ranges:

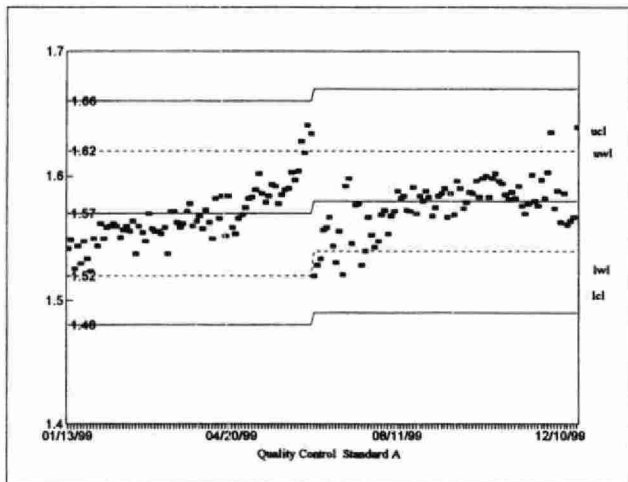
1.48	-	1.66	for	A (Jan-Jun)
1.49	-	1.67		A (Jul-Dec)
14.80	-	16.06	for	B (Jan-Jun)
14.82	-	15.96		B (Jul-Dec)
144.56	-	150.1	for	C (Jan-Jun)
145.01	-	150.4		C (Jul-Dec)
1287.7	-	1329.5	for	D (Jan-Jun)
1344.9	-	1458.4	for	D (Jul-Dec)

	n	Data Mean	Standard Deviation (1)
Stray Light	159	0.0378	0.0024

DUPLICATES:

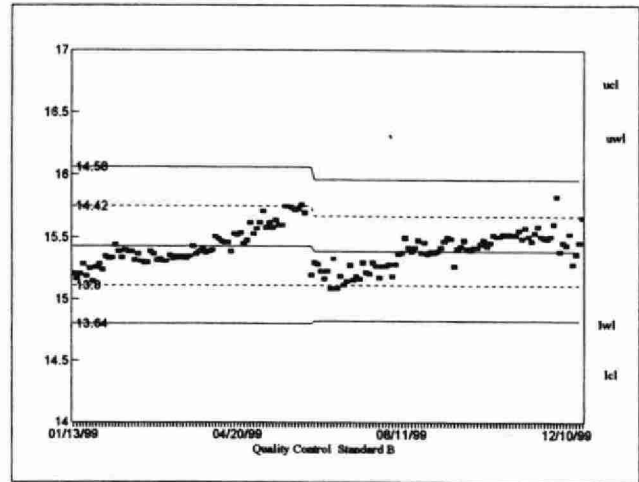
n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
246	0.0 - 2.0	0.0618	8.7
108	2.1 - 20.0	0.6299	9.3
29	21.0 - 200	2.3355	3.7
5	201 - 2000	7.0143	2.38
388	Overall	1.0750	9.93

TURBIDITY
QUALITY CONTROL DATA FROM 13/01/99 TO 30/12/99
E3311



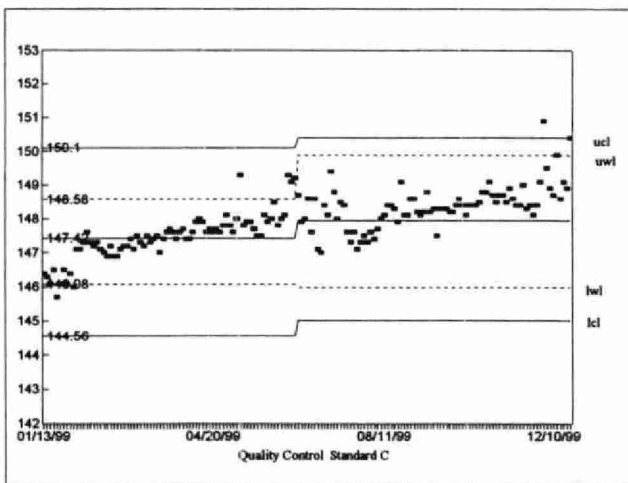
Jan - Jun

Jun - Dec



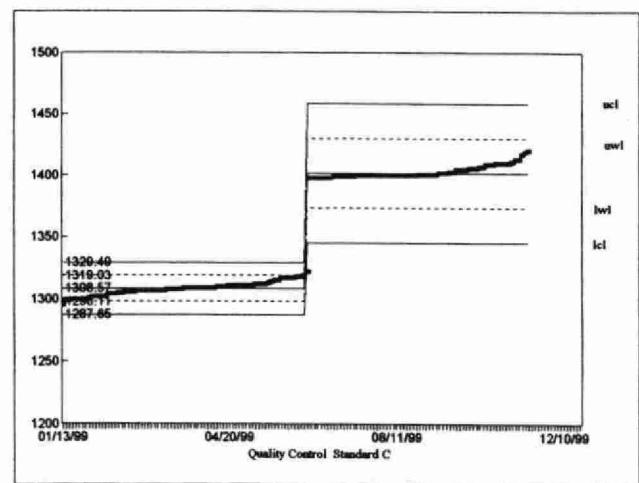
Jan - Jun

Jun - Dec



Jan - Jun

Jun - Dec



Jan - Jun

Jun - Dec

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ABBREVIATIONS

AAll	- Auto Analyzer Model II
AAS	- Atomic Absorption Spectrophotometer
Bl	- Blank
°C	- Degree Centigrade
cm	- Centimetre
CS1	- Check Sample 1
CS2	- Check Sample 2
Date	- Day/Month/Year
DO	- Dissolved Oxygen
EDTA	- Ethylenediaminetetra-Acetic Acid, Disodium Salt, Dihydrate
FTU	- Formazin Turbidity Units
g	- Gram
HZU	- Hazen Units
in ²	- Square Inches
IS(n)	- Internal Standard (n denotes parameter)
kg	- kilogram
L	- Litre
LAB	- Laboratory
LIMS	- Laboratory Information Management System
LTB/L	- Long Term Blank
lcl	- Low Control Limit
lwl	- Low Warning Limit
m ³	- Cubic Metre
M	- Molarity
MB	- Method Blank
meq	- Milliequivalent
mg	- Milligram
min	- Minute
mL	- Millilitre
mm	- Millimetre
N	- Normality
N.A.	- Not Available or Not Applicable
nm	- Nanometre
n	- Number
PC	- Personal Computer
Pure-DW	- Pure Deionized Water

ABBREVIATIONS cont'd

Pure-W	- Pure Water
QC	- Quality Control
QCA	- Quality Control Standard A
QCB	- Quality Control Standard B
QCC	- Quality Control Standard C
QCD	- Quality Control Standard D
R	- Recovery
rpm	- Revolutions Per Minute
RS92	- Reference Standard (in -house)
S	- Between Run Standard Deviation
S ₁	- Standard Deviation (Conventional)
S ₂	- Standard Deviation For Duplicates
S _w	- Standard Deviation Within Run
S. Class	- Weight Classification Designation (not certified)
s.d.	- Standard Deviation
Standard Cal	- Colourimeter setting to control electronic expansion
STD	- Standard
TCU	- True Colour Units
TPTZ	- Ferrous-2,4,6-tri(2'pyridyl)-1,3,5,- triazine
ucl	- Upper Control Limit
uwl	- Upper Warning Limit
µm	- Micrometer
µeq	- Microequivalent
µg	- Microgram
µS	- Micro-Siemen
UV	- Ultra-Violet
V/V	- Concentration based on volume measurements
W40	- Whatman 40 Filters
%	- Percent

Appendix A
W and T values for '99

Parameter	Method Reference No.	Units	Full Scale	W	T
Acidity, Gran	(E3248)	µeq/L H ⁺	1000	5.0	25.0
Acidity, Total Fixed Endpoint	(E3248)	mg/L CaCO ₃	1000	0.2	1.0
Alkalinity, Gran	(E3042)	mg/L CaCO ₃	-	-	-
Alkalinity, Total Fixed Endpoint	(E3042)	mg/L CaCO ₃	500	0.05	0.25
Alkalinity, Total Fixed Endpoint	(E3218)	mg/L CaCO ₃	1000	0.5	2.5
Aluminum, Total	(E3300)	µg/L Al	1000	2	10
Calcium	(E3249)	mg/L Ca	8.0	0.02	0.1
Carbon, Dissolved Inorganic	(E3028)	mg/L C	10.0	0.02	0.1
Carbon, Dissolved Inorganic	(E3370)	mg/L C	80.0	0.2	1.0
Carbon, Dissolved Organic	(E3370)	mg/L C	20.0	0.1	0.5
Chloride	(E3004)	µg/m ³ Cl	14.7	0.1	0.5
Chloride	(E3013)	µg/g Cl	-	0.5	2.5
Chloride	(E3016)	mg/L Cl	100	0.2	1.0
Chloride	(E3147)	mg/L Cl	2.0	0.01	0.05
Chloride	(E3148)	µg/filter Cl	2.0 mg/L	0.02 mg/L	0.10 mg/L
Chloride	(E3372)	mg/L Cl	1.0	0.01	0.05
Chlorine, Total Residual	(E3309)	µg/L Cl ₂	50	2	5
Chlorophyll "a"	(E3169)	µg/L	-	0.2	1.0
Chlorophyll "a" Acidified	(E3169)	µg/L	-	1.0	5.0
Chlorophyll "b"	(E3169)	µg/L	-	0.1	0.5
Colour, True	(E3025)	TCU	100	0.2	1.0
Colour, True	(E3219)	TCU	100	0.2	1.0
Conductivity	(E3024)	µS/cm	500	0.2	1.0
Conductivity	(E3177)	µS/cm	100	0.2	1.0
Conductivity	(E3218)	µS/cm	2000	1	5
Cyanide, Free	(E3015)	mg/L CN ⁻	0.2	0.001	0.005
		µg/g CN ⁻		0.01	0.05
Cyanide, Total	(E3015)	mg/L CN ⁻	0.2	0.001	0.005

Appendix A
W and T values for '99

Parameter	Method Reference No.	Units	Full Scale	W	T
Cyanide, Total	(E3015)	µg/g CN ⁻		0.01	0.05
Fluoride	(E3369)	mg/L F	2.0	0.01	0.05
Iron, Total	(E3303)	µg/L Fe	1000	2	10
Magnesium	(E3249)	mg/L Mg	2.0	0.005	0.025
Manganese	(E3303)	µg/L Mn	200	1	5
Nitrate	(E3004)	µg/m ³ NO ₃	14.7	0.1	0.5
Nitrilotriacetic Acid	(E3406)	mg/L NTA	1.00	0.01	0.05
Nitrogen, Ammonia Plus Ammonium	(E3364)	mg/L N	2.0	0.002	0.01
		µg/filter		0.05	0.25
Ammonia Plus Ammonium	(E3366)	mg/L N	50.0	0.05	0.25
Ammonia Plus Ammonium	(E3374)	µg/L N	1000	2	10
Nitrogen, Nitrate	(E3148)	µg/Filter N	2.0 mg/L	0.01 mg/L	0.05 mg/L
Nitrogen, Nitrate	(E3372)	mg/L N	1.0	0.01	0.05
Nitrogen, Nitrate Plus Nitrite	(E3364)	mg/L N	5.00	0.005	0.025
Nitrogen, Nitrate Plus Nitrite	(E3366)	mg/L N	50.0	0.05	0.25
Nitrogen, Nitrate Plus Nitrite	(E3374)	µg/L N	1000	2	10
Nitrogen, Nitrite	(E3364)	mg/L N	0.200	0.001	0.005
Nitrogen, Nitrite	(E3366)	mg/L N	2.00	0.005	0.025
Nitrogen, Total Kjeldahl	(E3116)	mg/g N	20	0.1	0.5
Nitrogen, Total Kjeldahl	(E3118)	mg/g N	100	0.2	1
Nitrogen, Total Kjeldahl	(E3367)	mg/L N	2.00	0.02	0.1
Nitrogen, Total Kjeldahl	(E3368)	mg/L N	50.0	0.05	0.25
Oxygen Demand, Biochemical	(E3182)	mg/L O	9.0	0.2	1
Oxygen Demand, Chemical	(E3170)	mg/L O	40.0	1	5
Oxygen Demand, Chemical	(E3246)	mg/L O	500	2	10
pH	(E3042)	-	-	-	-
pH	(E3218)	-	-	-	-
pH	(E3248)	-	-	-	-

Appendix A
W and T values for '99

Parameter	Method Reference No.	Units	Full Scale	W	T
Phenolics, Reactive	(E3179)	µg/L Phenol	50.0	0.2	1.0
Phosphorus,					
Reactive ortho-Phosphate	(E3364)	mg/L P	0.100	0.0005	0.0025
Reactive ortho-Phosphate	(E3366)	mg/L P	10.0	0.02	0.10
Phosphorus, Total	(E3036)	µg/L as P	range to 50 µg/L	0.2	1
Phosphorus, Total	(E3116)	mg/g P	5	0.02	0.10
Phosphorus, Total	(E3118)	mg/g P	8	0.02	0.10
Phosphorus, Total	(E3367)	mg/L P	0.200	0.002	0.01
Phosphorus, Total	(E3368)	mg/L P	10.0	0.02	0.10
Potassium	(E3249)	mg/L K	1.0	0.005	0.025
Silicon, Reactive Silicates	(E3370)	mg/L Si	10.0	0.02	0.10
Sodium	(E3249)	mg/L Na	4.0	0.005	0.025
Solids, Dissolved	(E3188)	mg/L	-	2	10
Solids, Suspended	(E3188)	mg/L	-	0.5	1.0
Solids, Suspended Ignited	(E3188)	mg/L	-	0.5	2.5
Solids, Total	(E3188)	mg/L	-	2.0	10.0
Solids, Total Ignited	(E3188)	mg/L	-	2.0	10.0
Sulphate	(E3004)	µg/m ³ SO ₄	14.7	0.1	0.5
Sulphate	(E3013)	µg/g		0.5	2.5
Sulphate	(E3147)	mg/L SO ₄	10.0	0.05	0.25
Sulphate	(E3148)	µg/filter SO ₄	10.0 mg/L	0.02 mg/L	0.1 mg/L
Sulphate	(E3172)	mg/L SO ₄	100	0.5	2.5
Sulphate	(E3372)	mg/L SO ₄	5.0	0.05	0.25
Sulphide, Hydrogen	(E3100)	µg/L H ₂ S		2.0	10.0
Turbidity	(E3311)	FTU	2000	0.05	0.25



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Janhurst, Susan

1999 performance

report : General *cms* akji

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